INFLUENCE OF SURFACTANTS ON THE STABILITY OF NANOFLUIDS OF RAW AND IMPREGNATED CARBON NANOTUBES FOR APPLICATION OF HEAT TRANSFER

BY

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Dedication to my beloved parents, my brothers, my sisters and my friend Abdallah Manasrah

ACKNOLODGMENT

In the name of Allah, the Most Gracious and the Most Merciful

All praise and glory be to Allah for his limitless help and guidance. Peace pleasing of Allah be upon his prophet Mohammed.

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LIST OF ABBREVIATIONS

Q	:	Heat transfer rate.
Re	:	Reynolds's number.
Nu	:	Nussle's number.
m_n	:	Mass flow rate for nanofluids.
Cp_n	:	Specific heat capacity of nanofluids
$T_{n,in}$	:	Tube inlet temperature.
$T_{n,out}$	:	Tube outlet temperature.
U	:	Overall heat transfer coefficient.
A	:	Cross sectional area of the tube.
ΔP	:	Pressure Drop.
GA	:	Gum Arabic.
PVP	:	PolyVinylPyrrolidone
SDS	:	Sodium Dodecyl Sulfate
DSC	:	Differential Scanning Calorimetry.
SEM	:	Scanning Electron Microscopy.
TGA	:	Thermo-Gravimetric Analysis.
MWCNT	:	Multi-Walled Carbon Nanotubes
n	:	Subscript for nanofluid.
ΔT_{lm}	:	Log mean temperature difference.
CTAB		Cetyl Trimethyl Ammonium Bromide

ABSTRACT

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In this study the effect of Poly Vinyl Pyrrolidone (PVP), Sodium Dodecyl Sulfate (SDS) and Gum Arabic (GA) surfactants on the stability and thermo-physical properties of CNT-water nanofluids were investigated. Raw and impregnated CNT with iron oxide were used for nanofluids preparation. CNTs were characterized using several techniques such as Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Thermo Gravimetric Analysis (TGA). Nanofluids were prepared using two step method at different CNT loading 0.1, 0.3, 0.5 and 1 wt% with different CNT:Surfactant ratio (1:1) and (1:0.5). The stability of nanofluids was tested in room temperature, under heating and mixing conditions and the ratio (1:0.5) was found to be stable for more than 6 months. Density, viscosity, thermal conductivity and specific heat capacity were investigated for the nanofluids by using all of the surfactants. The specific heat capacity was measured using Differential Scanning Calorimeter (DSC), the results show that thermal conductivity and specific heat capacity increases significantly with CNT loading and temperature while viscosity decreases with temperature increasing. Nanofluids prepared using SDS were found to have the highest viscosity and specific heat capacity. The heat transfer rate and the pressure drop for CNT-water with (1:0.5) GA surfactant were investigated by using

shell and tube heat exchanger. The heat transfer rate increased with increasing Reynolds also increased as the CNT loading is increased. The pressure drop was increased with CNT loading and the percentage of pressure drop increasing tend to decreases as the flow becomes more turbulent. The effect of doping iron oxide on the surface of CNT positively affected the heat capacity of GA nanofluids while this effect wasn't noticed for SDS and PVP nanofluids.

ملخص الرسالة

الاسم الكامل: اسماعيل وليد خليل المناصرة

عنوان الرسالة: تأثير المواد ذات النشاط السطحي على ثباتية الموائع التي تحتوي على جزيئات متناهية الصغر المكونة من أنابيب الكربون المتناهية الصغر الخامة والمطعمة بأكسيد الحديد وتطبيقاتها في المبادلات الحرارية.

التخصص: هندسة كيميائية

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تم في هذه الدراسة فحص تأثير ثلاثة أنواع من المواد ذات النشاط السطحي (البولي فينيل بوريليدون وكبريتات دوديسيل الصوديوم والصمغ العربي) على الثباتية والخصائص الحرارية-الفيزيائية للموائع التي تحتوي على جزئيات متناهية الصغر والمكونة من الماء وأنابيب الكربون المتناهية الصغر. تم استخدام أنابيب الكربون الخامة والمطعمة بأوكسيد الحديد في تحضير هذه الموائع. كما تم تشخيص الأنابيب الكربونية المتناهية الصغر باستخدام عدة تقنيات مثل المجهر الإلكتروني والمجهر الإلكتروني الإنتقالي بالإضافة إلى تقنية التحليل الوزني الحراري. وقد أعدت الموائع باستخدام التقنية الثنائية لتحضير الموائع المحتوية على جزيئات بأبعاد النانو وذلك باستخدام عدة تراكيز من الأنابيب الكربونية المتناهية الصغر وهي (0.1 و0.3 و0.5 و1 وزن%) كما تم إضافة المواد ذات النشاط السطحي بعدة نسب (كربون : المادة ذات النشاط السطحي) وكانت كالتالي (1:1) و (0.5:1). تم اختبار ثباتية الموائع واستقرارها على درجة حرارة الغرفة كما تم فحصها تحت تأثير التسخين والخلط. أظهرت النتائج أن العينات التي تم تحضيرها باستخدام نسبة (0.5:1) وجدت تكون ثابتة لأكثر من 6 أشهر باستخدام جميع التراكيز. كما تم فحص اللزوجة والكثافة والموصلية الحرارية والسعة الحرارية لمختلف العينات التي تم تحضيرها باستخدام الثلاثة أنواع من المواد ذات النشاط السطحي. تم قياس السعة الحرارية باستخدام جهاز الماسح التفاضلي لقياس السعات الحرارية. أظهرت النتائج زيادة كبيرة في الموصلية الحرارية والسعة الحرارية بزيادة تركيز أنابيب الكربون وزيادة الحرارة بينما اللزوجة كانت تتناقص بزيادة الحرارة. الموائع التي تم تحضيرها باستخدام كبريتات دوديسيل الصوديوم أظهرت أعلى قيم للسعة الحرارية واللزوجة. كما تم فحص التحسن في معدل انتقال الحرارة والزيادة في فرق الضغط باستخدام هذه الموائع في المبادلات الحرارية من نوع الغلاف والأنبوب. أظهرت النتائج زيادة واضحة في معدل انتقال الحرارة بازدياد رقم رينولدز وزيادة تركيز الأنابيب الكربونية كما لوحظ زيادة بسيطة في فرق الضغط باستخدام الموائع الجديدة. في الجزء الأخير تم فحص

تأثير تطعيم الأنابيب الكربونية بأوكسيد الحديد وذلك بفحص جميع الخصائص وأظهرت النتائج زيادة بسيطة في السعة الحرارية للعينات التي تم تحضيرها باستخدام الصمغ العربي أما الأنواع الأخرى فلم تتأثر النتائج.

CHAPTER 1

INTRODUCTION

1.1 Background

High performance cooling is one of the important requirements in many industries and technologies. The selection of heat transfer fluid for heat dissipation is an important consideration in the design of heat exchangers. Heat transfer fluids play a crucial parameter that affects the size and costs of heat exchangers. However, the available coolants like water and oils have low thermal conductivities which puts many limitations in the development of heat transfer to get the high performance cooling. The need for development new classes of fluids that enhance the heat transfer capabilities attracted the attention of many research groups over the world. In the last few decades' modern nanotechnology developed nanoparticles which have unique mechanical, electrical and thermal properties that could help the improving the heat transfer using nanofluids [1].

The suspended fluid with fine particles called "Nanofluid" which increases the heat transfer quality compared with the original fluid. Nanofluids are considered to be a new generation of heat transfer fluids and are considered to be two phase fluids of liquid-solid mixtures. The phrase "Nanofluid" refers to "Nano which mean the particle" and "Fluid which mean the suspension". Recently innovative materials which are nanometer sized have been used in common heat transfer fluids nanofluids and come to light as promising thermal fluids

for heat transfer application. The efficiency of the fluid could be improved by enhancing its thermal properties especially the thermal conductivity, and it is expected that the nanofluids will have a greater thermal conductivity than the base fluids [2, 3] . Adding micro sized particles to the base fluids have been done since 1960s [4]. But the problem of two phase suspensions that the sedimentation almost will happen for the micro suspended particles that leads to clogging the pipes and then the possibility of erosion of tube materials.

Many benefits could be noticed in nanofluids when compared with macrofluids, because of the huge specific area and smallest particle size, nanofluids can show superior characteristics like thermal conductivity, long term stability, clogging of channels, reduction in friction compared to base fluids [5], less pumping power, particle sedimentation by gravity and erosion of tubes.

Thermal and physical properties of single phase heat transfer fluids like water, oils and glycols are available in the literature and handbooks. But thermophysical properties of two phase nanofluids is still not explored extensively. In order to determine the heat transfer coefficients using nanofluids, an accurate measurement of the nanofluid properties is a prerequisite for these estimations. Then the suitability of nanofluids in heat transfer applications could be judged based on the heat transfer performance. Nanofluids considered to be a new generation of liquids for heat energy transport and can be employed as heat transfer fluids in heat exchangers in place of pure single phase fluids. Many applications could be included in the heat transfer improvement of nanofluids such as radiators in automobiles, solar water heaters, cooling of electronic devices and components in process industries and chemical engineering. The greatest importance of obtaining an

enhancement of heat transfer using nanofluids is to accommodate high heat fluxes and then reduce the size and cost of heat exchangers that results in the conservation of energy and materials.

In the last several years, many researchers performed a lot of work in order to develop heat transfer enhancement methods. Many additives have been used to improve the heat transfer properties of the base fluid. Therefore, the nanofluids will be probable to be perfectly used in the real applications even if their use may increase the pressure drop, and may change the heat transfer characteristics and transport properties of the fluid. Because of the fine nature of the nano particles, nanofluids took the behavior as a single phase rather than multi-phase.

There are many parameters affecting the thermal conductivity enhancement like the shape and size of the nanoparticle, the temperature of the fluid and its pH, the amount of nanomaterial added to the base fluid and the presence of any additives that could be added [6].

Carbon and its derivatives are the most interesting nanoparticles which are used in the area of heat transfer improvement using the nanofluids. The importance of these particles comes from the ultrahigh thermal properties of these materials compared with other materials as shown it table 1:1. Carbon nanotubes, graphite and graphene are the most well-known nanoparticles in this area. Graphene is a single sheet of graphite and after rolling this sheet it becomes nanotube “CNT”.

Due to the high thermal conductivity of these materials, it is expected that by suspending these particles into the base fluid, the nanofluid will have significantly higher thermal

conductivity compared with the base fluid. Many researchers worked to get the thermal conductivity of CNT and found it to be 3000 W/mK for multi wall CNT [7] and 3500 W/mK for single wall CNT [8]. The thermal conductivity of a single layer of graphene is 4840 – 5300 W/mK as reported by A.A. Balandin [9]. These high values of thermal conductivity opens the door to study the heat transfer improvement by suspending these particles into the traditional cooling fluids.

Generally, when researchers want to disperse or prepare nanofluids, they use sonication and homogenization technologies along the process to keep long time stability of the nanofluid. Few researchers used surfactants to have a stable fluid but it is not clear in the literature which surfactant is the best or for how long this fluid will be stable. These chemicals keep the nanoparticles suspended in the base fluid for a long time. Gum Arabic (GA), Polyvinylpyrrolidone (PVP) and Sodium Dodecyl Sulfate (SDS) are some examples of such chemicals.

To the best of our knowledge, the stability of CNT suspensions was not studied, so this work will focus on studying the heat transfer improvement of nanofluids and its stability for higher period of time using shell and tube heat exchanger. The nanofluids will be prepared by using sweet water to be near to the real application.

Table 1:1: Thermal conductivity of different solids and liquids [10].

Material	**Type**	**Thermal Condictivity (W/mK)**
Carbon	Nanotubes	1800 – 2000
	Diamond	2300
	Graphite	110 – 190
Metallic Solids	Copper	401
	Silver	429
	Aluminum	237
	Nickle	158
Nonmetallic Solids	Silicon	148
	Alumina	40
Metallic Liquids	Sodium	72.3
Nonmetallic liquids	Water	0.613
	Ethylene Glycol	0.253
	Engine Oil	0.145

1.2 Significance of Study

There are a lot of cooling fluids used in the industry such as ethylene glycol, water and mineral oils. These fluids have low thermal conductivity compared to solids. On the other hand, nanoparticles such as carbon nanotubes have ultra-high thermal conductivities. The idea comes by dispersing these nanoparticles into the base fluids to study the improvement of heat transfer using nanofluids. In the literature there is a lot information about dispersing CNTs in the base fluids but still these suspended fluids do not have a long stability, so different surfactants will be added to the nanofluid to increase its stability to allow studying the heat transfer improvement.

1.3 Problem Statement

This research focuses on studying the heat transfer improvement for CNT nanofluids in shell and tube heat exchanger taking into account different parameters such as the percentage of CNT, different types of surfactants and different chemicals impregnated on the surface of CNT. The stability of these nanofluids will be investigated as well.

1.4 Research Objectives

There are many objectives in this research:

1. To study stability of nanofluids using surfactants such as PVP, SDS, and Arabic gum. This objective will be done on three stages, first stage is investigating different percentages of surfactants, then testing how these nanofluids with surfactants

behave under higher temperatures and finally testing the behavior of the nanofluid under heating and mixing.

2. To impregnate the surface of CNTs with iron oxide nanoparticles with two concentrations 1 and 10 wt%.
3. To characterize CNT, physically mixed CNT and impregnated CNT using Thermo gravimetric Analysis (TGA), Differential Scanning Calorimeter (DSC), thermal conductivity analyzer and scanning electron microscopy (SEM) techniques.
4. To measure properties like density and viscosity for different concentrations of carbon nanotubes (0.1, 0.3, 0.5 and 1 wt. %).
5. To study the effect of physically modified and impregnated CNT and also unmodified CNT on the properties of the base fluid like thermal conductivity and specific heat capacity.
6. To investigate the heat transfer improvement in the shell and tube heat exchanger by using modified and unmodified CNT, this will be done at different flow rates of the cooling fluid, different weight percent of CNT and different types of modified CNT.
7. To study the effect of modified CNT (physically and impregnated) and unmodified CNT on the pressure drop along the tube inside the heat exchanger.

CHAPTER 2

LITERATURE REVIEW

The traditional way of enhancing the heat transfer in thermal processes is done by increasing the surface area of heat transfer, increasing the flow rate of the cooling fluid or dispersing solid particles in the fluid. However, there are many industries which require a new approach for enhancing the heat transfer such as transportation, electronics, chemical and petrochemical industry, energy supply plants and photonics industry [1].

Many types of particles were suspended in order to improve the heat transfer such as polymeric, metallic and non-metallic which tend to form slurries [10]. These slurries are sized from millimeters to micrometers could be a reason of many problems such as clogging of channels, erosion of pipelines and increasing the pressure drop in addition of instability and other rheological problems [11]. So these particles even they have high thermal conductivity there are not practical.

Nano particles have been firstly dispersed in the base fluids by a group of researchers in Argonne National Laboratory [10]. Choi was the first person who called the fluid which is suspended with nano particles "Nanofluid". Nanofluids compared with the fluids suspended with millimeter to micrometer particles show higher stability, thermal conductivity and rheological properties [12].

However, in the literature, less attention was given to stability of nanofluids while studying heat transfer improvement. It is very important to conduct studies on stability of nanofluids before performing experiment on heat transfer improvement in order to make it easy for

scale up to the commercial and industrial applications, as a result of that stability must be given more importance and attention. As stability of nanofluids could be improved, that will lead to better preparation which in turn will determine more concise and accurate measurement for thermo-physical properties like surface tension, viscosity, specific heat capacity, density and thermal conductivity. Enhancement of nanofluid stability also lead to better performance and efficiency in heat transfer utilities, also size reduction in heat transfer equipments, reduction in costs and better safety sides [5].

2.1 Preparation of Nanofluids

Generally in the literature, nanofluids consists of three constituents, base fluid, nanoparticle and a good dispersant or most of the publications without dispersant. Base fluids used by the reaseachers are water, engine oil, ethylene glycol, acetone and decane. Nanoparticles used are divided into four categories, metallic, oxides, carbon and nanodroplets while surfactants used are SDS, CTAB, Gum Arabic NADDNS and CMC.

Preparation process of nanofluids is an important issue in order to make sure that nanoparticles have a good dispersion within the base fluid. Masuda in 1993 obtained very poor stability of aluminum and titanium nanoparticles since particles tend to agglomerate [13]. Grimm 1993 also reported particle settling in his research [14]. Nowadays, stability of nanofluids has been further developed due to using surface active agents and innovate other nanoparticles that have high stability in the base fluids.

Preparation of nanofluid could be done by two methods, single step method and two steps method. Single step method, when the prepared nanoparticles remain in the same prepared solution. In this method, nanofluid will be the same solution used for preparing

nanoparticles. Eastman, Choi et al. in 2001 invented this technique using the process of physical vapor condensation method to produce Cu-ethylene glycol nanofluids. This process includes producing and dispersing the nanoparticles simultaneously in the fluid. The advantages of this process including the avoidance of drying, transportation, storage and a good dispersion of nanoparticles, that lead to minimize agglomeration and improving stability [15, 16]. Disadvantages and limitations of this process is the small sample size [13], low vapor pressure for these liquids that may vaporize at moderate temperatures and limitations on the particle size control [17]. The most well-known single step methods are "vacuum submerged arc nanoparticles synthesis" and the direct evaporation approach method [18].

The two step method is done by preparing the nano particles and nanofluid separately, and then nano particles are dispersed in the base fluid. This method is well known for nanofluid preparation. The process firstly includes the production of nanoparticles using chemical or physical methods, then these nanoparticles will be dispersed into the base fluid by sonication, magnetic force agitation, high-shear mixing, ball milling or homogenizing. Advantages of this method are large scale process and most economical than single step method. But unfortunately nanofluids prepared by this method are less stabile than single step method, this low stability could be enhanced by using surfactants. Nan et al. used a two-step technique with surfactants to stabilize CNT [19]. Hnog and Yang also used two step method in addition of ultrasonic disrupter to stabilize nanofluids [20, 21]. Most of the researchers whom are interested to use nanofluids in the application of heat transfer using two step method for preparation of nanofluids such as [22].

2.2 Stability of Nanofluids

There are two problems facing the nanofluids which are the agglomeration of nano particles and stability of the suspensions. These problems arise because there is no chemical affinity between base fluids and nanoparticles. Although nanofluids have better stability in the base fluids than microfluids, but still nanofluids have a high level of tendency to agglomerate because of the van der Waals forces between nanoparticles and that leads to sediment which in turn will result in poor improvement in thermal properties of nanolfuids. However, in order to have a long term stability for nanoparticles, there are two techniques that can be used to enhance stability and prevent agglomeration: the chemical dispersion technique and the physical dispersion technique.

These two methods used by the researches and they have a good result on long term stability. The physical dispersion technique includes the use of mehcnical fields such as ball milling, ultrasonic technology (Agitation) and the mechanical homogenization. Ultrasonic agitation includes the use of sonicators, in addition of the sonicator, additives could be used to enhance stability of nanofluids. Because of the high shear of ultrasonic technology, nanoparticles will be separated and the additives will provide enough repulsive forces to keep nanoparticles suspended in the base fluid for a long time and prevent sedimentation. The chemical dispersion method includes the use of chemical techniques such as the use of functionalization process by attach a chemical groups like COOH on the surface of the nanoparticles and that leads to damage of van der Waals forces between the suspended nanoparticles, but this method require additional steps and sometimes it needs much time and complex chemical reactions because the surface of the CNT is chemically

modified. The chemical dispersion method also includes the electrostatic technique which can be done by charge the nanoparticles with the same charge that leads to have a repulsive forces between the nanoparticles. The last method that sorted as a chemical dispersion method is steric dispersion which involves the use of surfactants. Surfactants are classified into four groups based on the charged on the head groups, nonionic, cationic, anionic and zwitterionic. For polar solvents it is better to select the water soluble surfactants, otherwise it is better to oil soluble surfactants. Generally it is preferable to use ionic (anionic and cationic) surfactants for CNT-water nanofluids. Using active surface agents will enhance stability of nanoparticles, using surfactants as a good dispersant still not studied well in the literature and many gaps still need to fulfill [5].

In the literature, different methods of dispersion have been used depending on the type of nanoparticle and the type of base fluid. Lee et al [12] used the oldest technique for dispersion, they used only the basic shaking and mixing to disperse aluminum oxide and copper oxide nanoparticles in ethylene glycol and also in water, but unfortunately in their results, they observe a large sedimentation and agglomeration for nanoparticles. Xwan and Li [23] studied the stability of copper-water and copper oil nanofluids, they used a physical dispersion method (ultrasonication) with the addition of Laurate salt and oleic acid as a dispersants; better results were obtained for copper-oil nanofluid that remains stable for seven days, but for copper-water nanofluid, agglomeration happened after 30 h. Estman [3] studied the vapor condensation method for copper particles and less sedimentation was observed in his study. L. Jiang and L. Gao worked to optimize the suitable percentage of SDS to get long term stability for CNT-water nanofluid, they found that the optimum quantity for 0.5 wt% of CNT is 2 wt% of SDS to get stable solution [24].

Patel [25] studied the thermal conductivity enhancement for gold and silver nanoparticles in water, in their research, they used citrate as a dispersant and they observed that citrate is a well dispersant for these nanoparticles. Xie [26] studied thermal conductivity enhancement for CNT-water and CNT-EG nanofluids, ultra-sonication technique with acid treatment was used to disperse CNT, they found that these nanofluids will be stable for two months.

Wen and Ding [27] worked on thermal conductivity of Al_2O_3-water nanofluids using SDBS as a surfactant and followed by 16-20 h of ultra-sonication The nanofluids that was prepared by this method remained stable for one week. Assael [28] studied the enhancement in thermal conductivity for CNT-water nanofluids using SDS and CTAB as surfactants used together as a dispersants followed by 24 - 490 min of ultra-sonication, they ended with a conclusion that the ultra-sonication time has significant impact on the stability of the prepared nanofluids. M. Liu and M. Lin [29] found that CNT-synthetic engine oil nanofluid with N-Hydroxy Succinimide (NHS) surfactant is stable enough to perform thermal conductivity measurements. Ding [30] studied the heat transfer improvement using CNT-water nanofluid, Arabic gum was used as a surfactant followed by ultra-sonication and high shear mixing. The prepared nanofluids remained stable for more than one month without any agglomeration. Jana [31] worked on the thermal conductivity enhancement for CNT and hybrid CNT. Only ultra-sonication was used to disperse the nanoparticles and the nanofluids were stable only for 30 min.

I. Madni and his group [32] investigated the effect of using mixed surfactants, anionic and cationic surfactants. They found that a 1:1 ratio between the cationic surfactant (Dodecyl trimethylammonium bromide DTAB) and the anionic surfactant (sodium octanoate SOCT)

with percentage equal to 20 dry mass based (20 Dmb%) is the optimum for concentration of 0.01wt% of MWCNT, and it is better than using 40 Dmb% of individual surfactant. Rashmi [33] studied the stability of CNT-water nanofluid and the impact of time required for sonication and the effect of concentration of Arabic gum, they concluded that 4 h of sonication will be enough to get a high stability with a concentration of 1 – 2.5 wt%, and these conditions can have a very stable nanofluid for more than 6 months.

Baku and Kumar [34], used ultra-sonication for 1 h to disperse CNT in water, nanofluids were stable for 7 days. Nasiri [35] disperse many types of CNT in water, SDS was use as dispersant followed by ultra-sonication, nanoparticles start to agglomerate after 400 h. Aravind [36] prepared MWCNT-water and MWCNT-EG to study thermal properties of these nanofluids, ultra-sonication for 40 min was only used to disperse nanoparticles, nanofluids were stable for 2.5 h for functionalized CNT and the functionalized shows the best dispersion. Nine [37] studied thermal properties for hybrid Al_2O_3-MWCNT-water nanofluids, ball milling and acid treatment were used to disperse nanoparticles in the base fluid, after 30 days about 23 – 25 % of the nanoparticles were agglomerated. Pham [38] studied the heat flux enhancement using Al_2O_3-CNT-water, Al_2O_3-water and Al_2O_3-water nanofluids, 1 h of ultra-sonication with the addition of boric acid was used to stabilize nanoparticles and the nanofluids were stable for 3 days.

The stability of nanofluids for a long time still is a challenge for the researchers because of the van der Waals forces that attracted particles to agglomerate and sediment in the bottom. It is very important to use a chemical dispersion technique (steric dispersion method) by the addition of suitable surface active agents followed by the ultra-sonication technology to make sure that the nanoparticles will be suspended in the base fluids for times as long

as possible. Also it is important to notice that if the unsuitable dispersion technique was used to disperse nanoparticles, nanoparticles clusters will be formed and it will agglomerate which in turn will decline the thermal properties directly [33].

2.3 Thermal Conductivity of Nanofluids

In order to study the heat transfer improvement using nanofluids, there are many thermo-physical properties required to be investigated such as density, specific heat capacity, thermal conductivity and viscosity. One of the most important aspects for the enhancement in heat transfer properties of nanofluids as coolants is the thermal conductivity. Consequently, many experiments have been conducted to investigate these properties. Studies that have been done in this area show that the enhancement in thermal conductivity affected by many parameters such as the concentration of nanoparticles in the base fluid, material of the nanoparticle used, type of the base fluid, the particle shape, temperature and particle size. In this research many other parameters will be added such as surfactant type, the concentration of the surfactant and impregnation of iron oxide with different concentrations doped on the surface of CNT.

As discussed in the previous section, the steric dispersion method for nanofluids that includes the use of surfactants followed by ultra-sonication is an effective method to have a long time stability. This method has two different views. In one hand R. Lotfi and A. Rashidi [39] said; although using surfactants like SDS and Arabic gum will enhance the stability of the nano solutions but unfortunately they have negative impact on the thermal properties of the nanofluid such as thermal conductivity. In the other hand D. Choi and J. Lee said that by comparing the results of using PVP as a surfactant, thermal conductivity

results are higher than without using surfactants. They explained these results because of the addition of surfactants such as PVP will improve dispersion and stability of the nanofluids which show higher values of thermal conductivity [40].

A. Chozatloo and A. Rashidi, studied the effect of surfactants on thermal conductivity. They studied different ways to disperse CNT in water as a base fluid. They measured the thermal conductivity and stability of the solutions. The maximum thermal conductivity was noted for treated CNT with a base (Potassium persulfate) and using SDS as a surfactant, they got up to 24.9 % enhancement in thermal conductivity at 0.1 wt% [41]. L. Chen and H. Xie [42] studied the effect of cationic Gemini surfactant which is chemically attached to the surface of CNT on thermal conductivity of the nanofluid. They found that the high concentration of Gemini surfactant has a negative impact on thermal conductivity and then sedimentation will occur.

In the literature the effect of particular materials with different concentrations on the thermal conductivity have been studied. All of the published work concluded that there is an enhancement in the thermal conductivity with increasing the concentration of nanoparticles. CNTs and graphene have a great interest in this application due to the high thermal conductivity compared with polymeric, metallic and non-metallic particles [43]. There is a lot of work done on nanofluids, which is mainly focused on the preparation techniques and measuring the thermal conductivity of the nanofluids. The highest thermal conductivity was shown by Choi [2], 150% increase in thermal conductivity by using 1% volume fraction of multi wall carbon nanotube dispersed in engine oil. This huge increase in the thermal conductivity was explained by Choi for two reasons, one is the high thermal conductivity of MWCNT and the high aspect ratio which was used.

Biercuk [44] studied two different nanofluids Single wall CNT-epoxy and vapor grown carbon fibers (VGCF)-epoxy, 125 % enhancement in thermal conductivity for SWCNT and 45% enhancement for VGCF nanofluid. Xie [26] studied the thermal conductivity enhancement using CNT-water, CNT-EG and CNT-Decene nanofluids, the maximum enhancement in thermal conductivity was for CNT-Decene nanofluid, up to 20% enhancement in k at concentration 1 vol%. It was concluded that a small amount of nanoparticles is enough to get high thermal conductivity values compared with the nanofluid. Assael [45] obtained 38% enhancement in thermal conductivity at 0.6 vol% for CNT-water nanofluid, SDS was used as a dispersant with 0.1wt%. Wen and Ding [27] studied CNT-water nanofluid, they obtained higher value for thermal conductivity, about 25% enhancement in k at 0.8 wt%. They concluded that the enhancement in thermal conductivity taking a linear relation then the results increased significantly and thermal conductivity increasing with temperature. Asseal [28] also studied the enhancement in thermal conductivity by dispersing nano particles in the base fluids. About 34% increase in thermal conductivity by using 0.6% volume fraction of MWCNT, also 8% increase in the thermal conductivity using 1% volume fraction of double wall CNT.

M. J. Assael and C. F. Chen studied the effect of SDS on thermal conductivity of MWCNT nanofluids at different percentages. They found that the addition of SDS to the nanofluid does not have any significant impact on the thermal conductivity, in the same research they found by FTIR that the hexagonal graphitic sheets did not affected by the addition of SDS and at the same time they found that the vibrations of the outside graphitic sheet affected by the addition of SDS, finally they concluded that SDS is the recommended surfactant for MWCNT [45]. While M. N. Patzali and A.A. Mouza found in their experiments that the

addition of cetyl trimethylammonium bromide (CTAB) surfactant has no effect on thermal conductivity measurements, they claimed that the reason is the different manufacturers of CNT and their properties [46]. Assael also tests the effect of CTAB and Nanosphere AQ on thermal conductivity for CNT-water nanofluid, it was found that there is a slightly decreasing in thermal conductivity in CTAB more than using nanosphere AQ but in the other hand samples prepared using CTAB were more stable than samples prepared using nansphere AQ surfactant [28].

Ding [30], studies the improvement in effective thermal conductivity for CNT-water nanofluid. Arabic gum was used as a surfactant with 0.25 wt% concentration, it was concluded that k increases with temperature and with increasing concentration of nanoparticles.

Liu [29] studied the enhancement in k for CNT-EG and CNT-synthetic engine oil nanofluids, 12.4% improvement in k for 1 vol% of CNT-EG and 30% enhancement for the seond nanofluid at concentration of 2 vol%. Hwang [47] studied different types of nanoparticles, the maximum enhancement was for CNT-water nanofluid, 11.3% enhancement in k was obtained for 1 vol% concentration.. Hwang [48] studied CNT-water and CNT-oil nanofluids, 7% and 8.7% enhancement for CNT-water and CNT-oil at a concentration of In the review, publications related to carbon derivatives will be shown in details. % and % respectively.

Amrollah [49] obtained 20% enhancement in thermal conductivity for 2.5% concentration of CNT-EG nanofluid. Carg [50] used 0.25% of Arabic gum as a surfactant followed by 40 min ultra-sonication for CNT-water nanofluid, the maximum enhancement in k was 20%, it was concluded that further sonication more than 40 min will have negative effect

on thermal conductivity measurements. Amrollah [51] again studied the thermal conductivity for single wall CNT-water with the addition of Arabic gum and SDS as a dispersants, 30% and 35% enhancement in k at 1 wt% (at 30 °C) and 0.5wt% of CNT respectively, it was concluded that the optimum concentration of Arabic gum is 0.1 wt% for 0.5 wt% of CNT nanoparticles. Teng [52] studied carbon-water nanofluid, the maximum enhancement in k was 25% at 50 °C. Rashmi [33] obtained a huge increase in the thermal conductivity for CNT-water nanofluid for nanoparticles concentration was 0.01 – 0.1 wt% and concentration of Arabic gum was 1.0 – 2.5 wt%. It was obtained that 100 – 250 % increase in thermal conductivity in temperature range 25 – 60 °C, KD-2 prothermal conductivity meter was used to measure thermal conductivity. Baby [6] studied graphene-water and graphene EG nanofluids and 75% enhancement in thermal conductivity was reported using DI water at 50 ºC.

Kumaresan and Velraj [53] studied a different type of base fluids (water-EG mixture) suspended with CNT nanoparticles, there measurements was done at 40 °C, the maximum enhancement in thermal conductivity was 17.45% at a concentration of 0.45 vol%. Ruan and Jacobi [54] studied CNT-water and CNT-EG nanofluid, the same enhancement was obtained for both of the nanofluids, 9% enhancement in thermal conductivity at 0.24 vol% concentration.

Kumaresan [55] studied another mixture (water-EG) with CNT nanoparticles, the maximum enhancement in thermal conductivity was 17.45% at 0.45 vol% and 40 °C. Park and Kim [56] studied the enhancement in thermal conductivity for different types of graphene in distelled water, 5.47% and 4.45% enhancement in k for 6.24 and 0.01 vol%. Ma [57] obtained 18.9% enhancement in thermal conductivity at 0.07 wt% of

functionalized graphene. Kole and Dey [58] studied functionalized graphene suspended in distelled water, enhamncement up to 15% for 0.395 vol%.

Helefadl [59] obtained 40, 70 and 85 enhancement in thermal conductivity coefficient C_k for CNT-water nanofluid at 20, 30 and 40 °C, respectively. Chozatloo and Rashidi [41], also studied the effect of surfactants on thermal conductivity. They studied different ways to disperse CNT in water as a base fluid. They measured the thermal conductivity and the stability of the solutions. The maximum thermal conductivity was noted for treated CNT with a base (Potassium persulfate) and using SDS as a surfactant, they got up to 24.9 % enhancement in thermal conductivity at 0.1 wt%.

The mentioned literature about the enhancement in thermal conductivity was only taking care of the carbon derivatives nanofluids. Many researchers worked on other nanoparticles such as CuO and Al_2O_3 by [12, 60-70], Cu [3, 23, 63], gold and silver [25, 71], TiO_2 [22, 68-70, 72-74], Al_2Cu and Ag_2Al [75], SiO_2 [66], SiC [76], Fe [68, 77].

Material of the nanoparticles affect the heat transfer improvement and thermal conductivity enhancement. Hwang [47] compared the thermal conductivity of many types of nanoparticles, the highest thermal conductivity was for CNT because of the highest thermal conductivity of CNTs. Choi [2] obtained a huge increase in thermal conductivity due to the superior properties of CNT and the highest L/D ratio that means a large amount of heat could be transported within the nanotubes which in turn means higher thermal conductivity values.

The base fluid have an important role in the enhancement of thermal conductivity. It was concluded by Rashmi [5] that the highest thermal conductivity of the base fluid the lowest

enhancement in thermal conductivity. Wang [67] studied different types of base fluids namely EG, water and vacuum pump oil suspended with CuO and Al_2O_3 nanoparticles. The highest k was for Al_2O_3-EG and the enhancement in k was high for EG then water then engine oil and finally vacuum pump oil. Xie [26] dispersed CNT in water, decene and EG. The highest enhancement was for pump oil then EG and then water. Recently, Ruan and Jacobi [54] reported same results. From the previous results in addition of Liu [29], it can be concluded that by increasing the thermal conductivity of the base fluid the thermal conductivity of the nanofluid will be decreased.

The particle size has an important effect on the thermal conductivity, it was observed by many researchers that smaller particle size will have higher thermal conductivity because of the high surface area and due to the Brownian motion and liquid layering. This trend was obtained by [67, 76-78]. Particle shape also has an important effect, it was concluded that particles have larger surface area will have higher thermal conductivities and then rapid energy transport, this observations concluded by Murshed and Xie [22, 76].

Almost, the effect of temperature have been studied by all of the researchers, it can be concluded that by increasing the temperature, thermal conductivity will increase. According to Wen and Ding [27] it can be concluded that the improvement in thermal conductivity will be higher at high temperatures.

The enhancement of thermal conductivity of nanofluids because of the high thermal properties of nanoparticles was attributed in many works to the Brownian motion, which is the irregular motion of the suspended particles and the zig zag motion due to molecular collisions between the suspended nanoparticles. Keblinski [79], Xuan [80] supported the idea of the role of the Brownian motion and Xuan developed a model for effective thermal

conductivity based on the Brownian motion. Jang and Choi [81] claimed that the Brownian motion have a great importance in the nanoscale convection and the improvement in heat transfer.

Other factors may affect thermal conductivity such as pH and additives. Xie [76], Lee [82] and Wang [83] studied the effect of pH on thermal conductivity for different nanoparticles but finally it was concluded that the effect of pH does not strongly affected the thermal conductivity. Rashmi [5], concluded in her review paper that still the effect of additives need to be investigated by preparing samples with additives only without nanoparticles and then with nanoparticles.

2.4 Viscosity of Nanofluids

The second thermo-physical property that was studied is the viscosity. The viscosity of nanofluids did not gather much attention like thermal conductivity, but yet it is an important parameter that requires an attention to make easy to scale up nanofluids to the commercial scale. Thermal conductivity was affected by the viscosity of the nanofluid which was done by Eastman, Keblinski and Das [84-86]. It was concluded that the viscosity have a negative effect on the thermal conductivity. In order to transfer the world of nanofluids to the industry and commercial scale, high thermal conductivity with low viscosity is rewuired, here the importance of our research appears.

Generally, many parameters affected the viscosity of nanofluids such as concentration of nanoparticles, additives like surfactants, concentration of the surfactants, temperature and shear rate. For CNT nanofluids, Ding [30] studied the viscosity of CNT-water nanofluid,

it was concluded that the viscosity increases with increasing the concentration of CNT and decreases with increasing temperature. Ruan and Jacobi [54] studied CNT-water and CNT-EG, the maximum increasing in viscosity was 30% for 0.24 vol% of CNT-EG nanofluid. Kumaresan [55] studied a mixture of EG-water suspended with CNT, the increase in viscosity was 0.4%, 0.22% and 0.08% for a concentration of CNT 0.45 vol%, 0.3 vol% and 0.15 vol % at 20 °C, respectively. Halffadl [59] studied water-CNT nanofluid, it was concluded in his work that the viscosity decreased with increasing in shear rate. Saleh [72] and Das [87] concluded that viscosity of nanofluids increased with increasing particle size. Saleh [72] and Fadele [73] concluded that viscosity is independent on temperature, but Duangthongsuk [74] and Li [88] observed that by increasing temperature the viscosity will decrease. Duangthongsuk [74] and Wang [67] also observed that by increasing the concentration of nanoparticles the viscosity of nanofluids tend to increase. Other researchers' works to obtain models to measure thermal conductivity and there are listed in [5]. But still viscosity of nanofluids need to be investigated and the effect of dispersants required deep studies to transfer this technology to the commercial scale.

2.5 Density of Nanofluids

Like the viscosity, density did not gain much attention by researchers. Density is one of the parameters that affect the heat transfer characteristics of nanofluids. Generally, because of the low concentrations of nanoparticles used in preparation nanfluids, there is a negligible increasing in density compared with the base fluid. Rashmi [33] concluded the same result that the increasing in density is negligible. Teng [52] studied the density of carbon-water nanofluid, the density increased up to 0.39% for 0.02 wt% at 50 °C. Vajjha

[89] studied different types of nanoparticles suspended in water and EG with high concentrations, it was concluded that by increasing the concentration of nanoparticles, density will increase. Pak and Cho [90] proposed an equation to calculate the density of nanofluids but this equation taking into account the density of base fluid, density of nanoparticles and volume fraction of the nanoparticles but concentration of dispersant and dispersant type are not included. Density when compared with other parameters, like thermal conductivity, viscosity and specific heat capacity is less important due to the negligible increasing in density at the used concentrations of nanoparticles.

2.6 Specific Heat Capacity of Nanofluids

The specific heat capacity is very important like thermal conductivity. Thermal conductivity taking care of the heat transport and specific heat capacity is considered to be a thermodynamic property to express the physical meaning of heat storage. There is no published work related to calculate the specific heat capacity experimentally, the published data either simulated or using Pak and Cho [90] correlation, this correlation same as the viscosity correlation it is taking into consideration specific heat capacity of base fluid, specific heat capacity of nanoparticles and volume fraction of the nanoparticles. Still there is a huge gap in specific heat capacity measurements. DSC is a good equipment to estimate heat capacity but no one used that. In this research calculating the specific heat capacity of the nanofluids is one of the main objectives.

2.7 Convective Heat Transfer of Nanofluids

Applying nanofluids as a coolant liquids in heat transfer equipment have been carried out in many studies. Heat transfer equipment that used for the application of heat transfer are circular tubes, heat exchangers and circulation flow systems. To express the improvement in heat transfer improvement, many parameters are used to calculate that enhancement such as heat transfer rate, convective heat transfer coefficient, heat flux and Nusslte number. In the literature also the type of the flow (turbulent or laminar) was considered by many researchers.

Ding and Alias [30] studied the effect of MWCNTs on the heat transfer performance of nanofluids using a straight copper tube. They found that the highest enhancement of the convective heat transfer coefficient was 350% for 0.5 wt. % of CNT at an 800 Reynolds number. They explained this enhancement by suggesting possible reasons. For example, the CNTs have high aspect ratios, the presence of nanoparticles in the thermal boundary layer may be reduced, and the re-arrangement of particles, and shear-induced thermal conduction enhancement could all be explanations.

Carg [50] used CNT-water nanfluid with 0.25 wt% of Arabic gum as a dispersant, a straight tube made from copper was used to take measurements. The maximum enhancement for convective heat transfer coefficient was 32% at Reynolds 600 ± 100. Halelfadl [59] also used CNT-water nanofluid and it was recommended that the efficiency of the nanofluid increased when Re increased. Kumar [91] disperse graphene in a mixture of water-EG, a simple heat exchanger was used in his study, the maximum enhancement for heat transfer coefficient was 40% at 0.8 vol%, it was concluded that at higher values of Re, the

enhancement in heat transfer coefficient will be higher. Mare [92] used a plate heat exchanger with CNT-water and Al_2O_3-water nanofluids, the experiments was done in the laminar region, the maximum enhancement in heat transfer coefficient was 50% for CNT-water nanofluid. Lotfi [39] used shell and tube heat exchanger with CNT-water nanofluid, it was concluded in the work that the presence of CNT in the nanofluid will increase the heat transfer rate. Ghozatlo [41] dispersed graphene in water the convective heat transfer coefficient was increased up to 35.65% at 0.075 wt% and 38 °C. Choi [40] investigated the enhancement in Convective HTC for graphene, graphite and CNT suspended in stearic acid, 3.35 times enhancement in HTR using graphite at 5 vol%.

There are different nanofluids inserted to heat transfer equipment. Different heat transfer improvement for each nanoparticles. For shell and tube heat exchanger, Farajollahi [93] used Al_2O_3-water and TiO_2-water nanofluids in shell and tube heat exchanger, it was concluded in his work that by increasing Peclet number the heat transfer properties will increased significantly and the enhancement in heat transfer is higher while using small or higher concentration of nanoparticles. Leong [94] also used shell and tube heat exchanger with Cu-EG nanofluid, 7.8% enhancement in convective heat transfer coefficient using 1% of Cu.

Other studies that have been performed to study the enhancement in convective heat transfer coefficient used other nanoparticles such as titanium oxide, aluminum oxide and zinc oxide with different base fluids such as water, oil and EG. The same trend was noticed in all publications that by increasing the concentration of nanoparticles the heat transfer characteristics will increase.

Generally, using nanofluids in the heat transfer equipment will directly inhance the convective heat transfer coefficient in addition of Nusselt number. Improvement in heat transfer coefficient is dependent on the kind of the heat transfer process that used in experimentations. As the previous properties discussed in the last sections, the convective heat transfer coefficient depend on many factors such as the concentration of nanoparticles, viscosity of the nanofluid, temperature and type of dispersant. Many researchers worked to formulate models for effective heat transfer coefficient which are listed in [5].

Pressure drop is one of the most important parameters that must be studied in the heat transfer equipment. Pressure drop is directly related to viscosity that by increasing viscosity of the nanofluid, pressure drop will increase and then larger pumping power and more cost. Vasu [95] observed that pressure drop within the heat exchanger is doubled compared with the base fluid at 4% concentration. Panzali [46] reported that there was 40% increase in pumping power for nanofluid more than for the base fluid. This factor also need more investigation and still not fully covered in the literature.

2.8 Mechanisms of Heat Transfer for Nanofluids

Enhancement in thermal conductivity for nanofluids was explained in the literature by several mechanisms. Brownian motion, liquid layering and particle clustering are the most mechanisms that could be the reason for that enhancement. Koo [96] and Shukla [97] concluded that the Brownian motion has a significant effect on the enhancement in thermal conductivity, but Kelbenski [79] assumed that the Brownian motion could be neglected as a reason for the enhancement in thermal conductivity compared with thermal diffusion within the particles. Jang and Choi [98] explained the enhancement in thermal conductivity

due to the collisions between the base fluid molecules, the collisions between the nanoparticles itself which is the Brownian motion, thermal diffusion within the particles and thermal interaction between the suspended nanoparticles and base fluid molecules, Choi concluded that Brownian motion has no significant impact on the thermal conductivity enhancement when compared with other reasons. Prsher [99] added other mechanisms related to Brownian motion that could explain the enhancement in thermal conductivity such as the interparticle potential and the liquid convection that caused by the Brownian motion.

Another mechanism that could explain the enhancement in thermal conductivity is the nanolayer. Nanolayer is a structure that formed between the nanoparticles and the surface of the base fluid molecules. Keblenski [79] said that nanolayer will work as a thermal bridge between nanoparticles and base fluid molecules that leads to thermal conductivity enhancement. However, the explanation of nanolayer to be a reason for k enhancement have some contradictions and it was used to create a models for thermal conductivity.

Nanoclusters is the third possible mechanism that could explain the enhancement in thermal conductivity. Nanoclusters leads to enhance thermal conductivity but in the other hand nanoclusters means increasing in mass which in turn will lead to sedimentation because of the increasing in mass. Yu [100] studied the effect of temperature on clustering, it was observed that by increasing temperature clustering tend to decrease and that may explain the significant increase in thermal conductivity at higher temperatures.

However, still the enhancement in thermal conductivity and convective heat transfer coefficient needs more investigation and deep understanding about relations between nanoparticles.

CHAPTER 3

METHODOLOGY

It was obtained from the literature that nanoparticles have enhanced the thermo-physical properties of the base fluids. Convective heat transfer coefficient can be enhanced up to 250% and thermal conductivity increased with different ratios according to the method used for measurements. However, preparation of nanofluids with long time stability is still a big challenge. The dispersant will have the highest enhancement in thermal conductivity and specific heat capacity with lower viscosity is also sill a challenge. Which dispersant will provide the best interaction between nanoparticles and base fluid molecules? Does nanofluids have a good heat transfer enhancement in shell and tube heat exchangers? What will be the pressure drop in the heat transfer equipment using nanofluids? What is the optimum conditions of nanofluid, such as the concentration of the dispersant and concentration of CNT nanoparticles that will be the best for operating heat transfer equipment? Is there any effect of the surface modification of CNT with dispersant on the heat transfer characteristics? What is the effect of aspect ratio with dispersant on the heat transfer properties? All of these questions will be discussed in this work.

This chapter will discuss the methods and materials that will be used in preparation, stability, characterization and implementation the CNT-water nanofluid. The materials used in the research such as the CNT, dispersants and the metal nitrates that used for impregnation. The effect of dispersants on stability of nanofluids and impregnation process that includes the surface modification of CNT will be discussed. In Characterization of

nanofluids will discuss the improvement in thermo-physical properties of nanofluids in addition of surface modification of CNT. Implementation will handle the application of nanofluids in the shell and tube heat exchanger and studying other parameters such as pressure drop.

3.1 Materials

Different materials will be used in preparation CNT-water nanofluids. Basically this study is based on the heat transfer enhancement using CNT nanoparticles. The commercial multi wall carbon nano tubes (MWCNTs) purchased from Chengdu Organic Chemicals Co. Ltd. "TimesNano", China with the following specifications: 10 to 20 nm outer diameter with length from 10 to 30 μm, 200 m^2/g specific surface areas and 95% MWCNT purity.

Iron nitrate will be used to modify the surface of CNT nanoparticles. Iron oxide will be attached on the surface of CNT with different concentrations (1wt% and 10wt%). Iron nitrate was chosen based on the results of previous research that was done by Abdallah manasrah [104], it was concluded in his work that iron oxide increases thermal properties of CNT-water nanofluid when it attached on the surface of CNT and the iron oxide has a better results than copper oxide and aluminum oxide. Iron nitrate $Fe(NO_3)_3.9H_2O$ (Mol wt 404 g/mol) used as a precursor in impregnation process manufactured by LOBAL Chemie, with > 98% purity.

Different surfactants will be used in this study in order to achieve most of research objectives which are gum Arabic from aciacia tree, SDS (> 99% purity) and PVP (average mol wt 10,000). All of them are commercially imported from SIGMA – ALDRICH.

Sweet water will be used as be base fluid, properties of this water will be the reference in all of the characteristics of the nanofluid.

3.2 Nanofluid Preparation

Nanofluid preparations consists two main steps, surface impregnation and nanofluid preparation. Nanofluid preparation includes the preparation of the unmodified CNT and impregnated CNT.

3.2.1 Impregnation of MWCNT with iron oxide nanoparticles

Iron oxide nanoparticles will be doped on the surface of CNT by impregnation method. Iron nitrate manufactured from Sigma and Aldrich with purity 99.9% will be used as a precursor to produce the metal oxide on the surface of CNT. The impregnation process is a simple process done firstly by choosing the percentage of iron oxide to be loaded on the surface of CNT. The estimated quantity of iron nitrate will be immersed in alcohol such as ethanol in this case study, then the required quantity of CNT will be immersed in the same container. After that the overall solution must be sonicated using ultra-sonication technology that depends on sound waves helping to distribute CNT nanoparticles from each other and also have an important role to homogeneously distributing the iron nitrate within CNT particles. In addition of that the main objectives of ultra-sonication process to reduce the agglomeration of CNT and increasing dispersion level of nanoparticles in order to have maximum contact time between CNT and iron nitrate also to have a complete and homogeneous wetting of the whole sample particles during impregnation. Ultra-sonication process done at room temperature and continued form 30 – 45 min, which is enough time

to distribute CNT particles and as discussed in the literature, many researchers concluded that this is the optimum time to have a good stability for nanofluid.

It is very important to immerse CNT and iron nitrate in an enough amount of alcohol. Low quantity tend to decrease dispersion of nanoparticles and prevent iron nitrate to reach to all CNT nanoparticles. On the other hand large quantity my lead to separate both of the constituents which in turn will decrease quality of the impregnation process. Another important note for ultra-sonication process is to select an optimum frequency of sonication which depends on many factors such as the concentration of nanoparticles, size of the beaker used for ultra-sonication and amount of alcohol added to impregnation process.

After finishing ultra-sonication process, sample must be put in an oven at a temp of 60 °C for 12h or more in order to evaporate alcohol and dry the sample as shown in figure 3.1. Finally, the samples were subjected to calcination step at 350 °C for 4 hr to produce iron oxide nanoparticles on the surfaces of carbon materials.

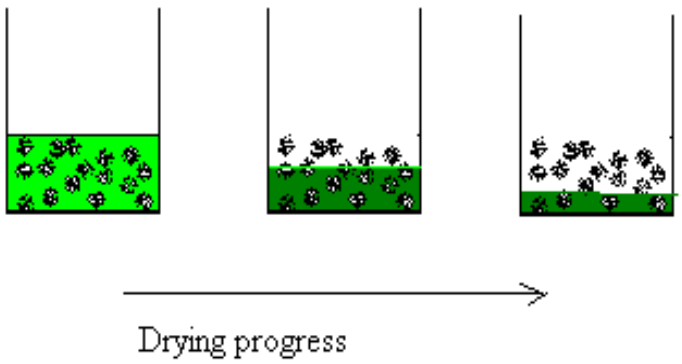


Figure 3:1: Schematic diagram explains drying process for impregnation process

3.2.2 Preparation Stage

All of nanofluid samples that prepared for characterization or for the application in the shell and tube heat exchanger will be prepared according to this procedure. From introduction chapter there was two methods for preparing nanofluids, two step method was selected to prepare nanofluids for this study. The two step method includes preparing nanoparticles and then suspending them in the base fluid. Firstly the selected mass concentrations of the CNT and surfactant must be weighted and immersed in the required quantity of water. Sweet water will be used for this study.

After having all of the constituents in one beaker, the sample then transferred to ultra-sonication equipment to sonicate the sample and produce the well dispersed homogeneous nanofluid. The ultrasonic pulses of 750 W and 20 kHz generated by an ultrasonic cell disrupter shown in Figure 3.2 was used to improve the dispersion of CNT in the base fluid.

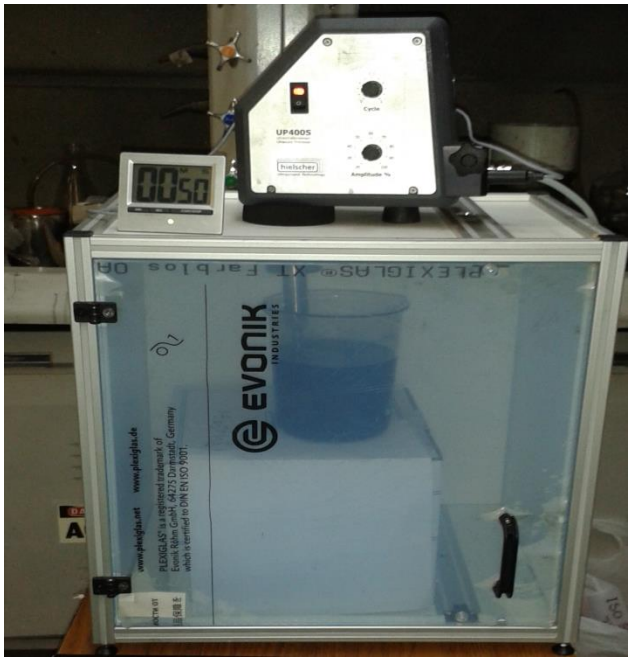


Figure 3:2: Ultrasonic cell disrupter (Sonicator)

3.3 Stability of Nanofluids

In this part, stability of the nanofluids will be tested at different concentrations of nanoparticles and different concentrations of surfactants. Different concentrations were selected for nanoparticles 0.1, 0.3, 0.5 and 1 wt%. Three surfactants were added to the solutions (gum Arabic, SDS and PVP) at three different ratios (CNT: Surfactant), (1:1), (1:0.5) and (1:0.2).

Stability will be done on three stages, the first stage includes how much time does the samples remains stable without agglomeration and sedimentation at the bottom. This stage only including preparing samples, leave them under room temperature and observing what will happen to the samples with time. The second stage preparing samples and study the effect of mixing on the samples, this stage done by placing samples on magnetic stirrer, keeping stirring and noticing what will happen to samples (agglomeration, sedimentation or evaporation of water). The last stage includes the double effect of heating and stirring, also this step done by preparing samples and keeping heating and stirring to observe what will happen with time and register the observations.

3.4 Characterization of Nanofluids

Characterization will be done to investigate that the impregnation process was done successfully and to characterize the thermos physical properties of nanofluids. Investigation that the impregnation process have been done successfully done by testing weight loss with temperature using thermos gravimetric analysis (TGA), testing under

microscope using scanning electron microscopy (SEM) and testing the constituents of the sample using Energy Dispersive X-ray analysis (EDX).

The second part of characterization is to characterize the thermos physical properties of nanofluids. This includes nanofluids prepared from the impregnated CNT and the unmodified CNT. The aim of studying thermos physical properties is to study the effect of surfactants on these properties and to come out with a conclusion that one of the surfactants is recommended to be used in the real applications. Characterization of thermos physical properties includes the measurements of density, viscosity, specific heat capacity and thermal conductivity.

3.4.1 Thermal Gravimetric Analysis (TGA)

Thermo gravimetric analysis is an equipment will be used to study the thermal stability for a material. This test depends on the loss of sample weight with heating. Measurements in this equipment done using inert gas such as argon and it will record the weight loss as a function of temperature. The Thermo gravimetric analysis can provide an information about thermal stability, the composition of multi component samples, decomposition kinetics, oxidative stability, calculated life time of any product and moisture and volatile component of a material. The equipment consist of a crucible which holds the sample, very sensitive balance which continuously monitoring the sample weight and furnace which used to heat the sample to high temperatures in order to perform the test. TGA results includes a curve between the change in sample weight “in percentage of the original weight on Y-axis” and the material temperature on X-axis.

A Netzsch model STA 449 F3 Jupiter® TGA was used in this study gravimetric analysis. The measurements of weight loss were taken from 25 to 1400 ^{o}C with 20^{o}C/min increment temperatures. Argon was used as the inert gas. The used TGA shown in figure 3.3.

Figure 3:3: Photograph of Netzsch model STA 449 F3 Jupiter® device.

3.4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a familiar technology will be used for visualizing and analyzing surface morphology and micro structural properties of samples.

This equipment works by producing a range of signals on the surface of samples using focused beam with high energy electrons. These signals that come from electron sample

interactions illustrate the sample information such as crystalline structure, surface morphology and chemical composition.

In general the sampler will collect the data over a selected area on the surface of the sample, and then the result will be 2D image which displays the spatial variations in the surface characteristics of the sample.

In SEM, a sample is exposed by an electron beam and data on the sample are transported by secondary electrons emitted from the surface layer of thickness ~ 5nm and by backscattered electrons released from the volume of linear size ~ 0.5μm. SEM equipment shown in figure 3.4.

3.4.3 Energy Dispersive X-ray analysis (EDX)

Energy dispersive X-ray spectrometer (EDX) is attached equipment to the scanning electron microscope which analyzes the X-ray radiation emitted from the sample when the electron beam reacts with the sample. The main objective of using EDX to investigate the composition of the sample accurately. After exposing specimen to high energy electron beams, different atoms appears in the sample which emit characteristic X-rays which could be represented as multiple distinct peaks on the energy scale. The intensities of the peaks will be compared with the peaks of the standard sample to get the amounts of each atomic species, then the accurate composition of the specimen could be calculated. This equipment attached with SEM equipment and figure 3.4 shown the same equipment.

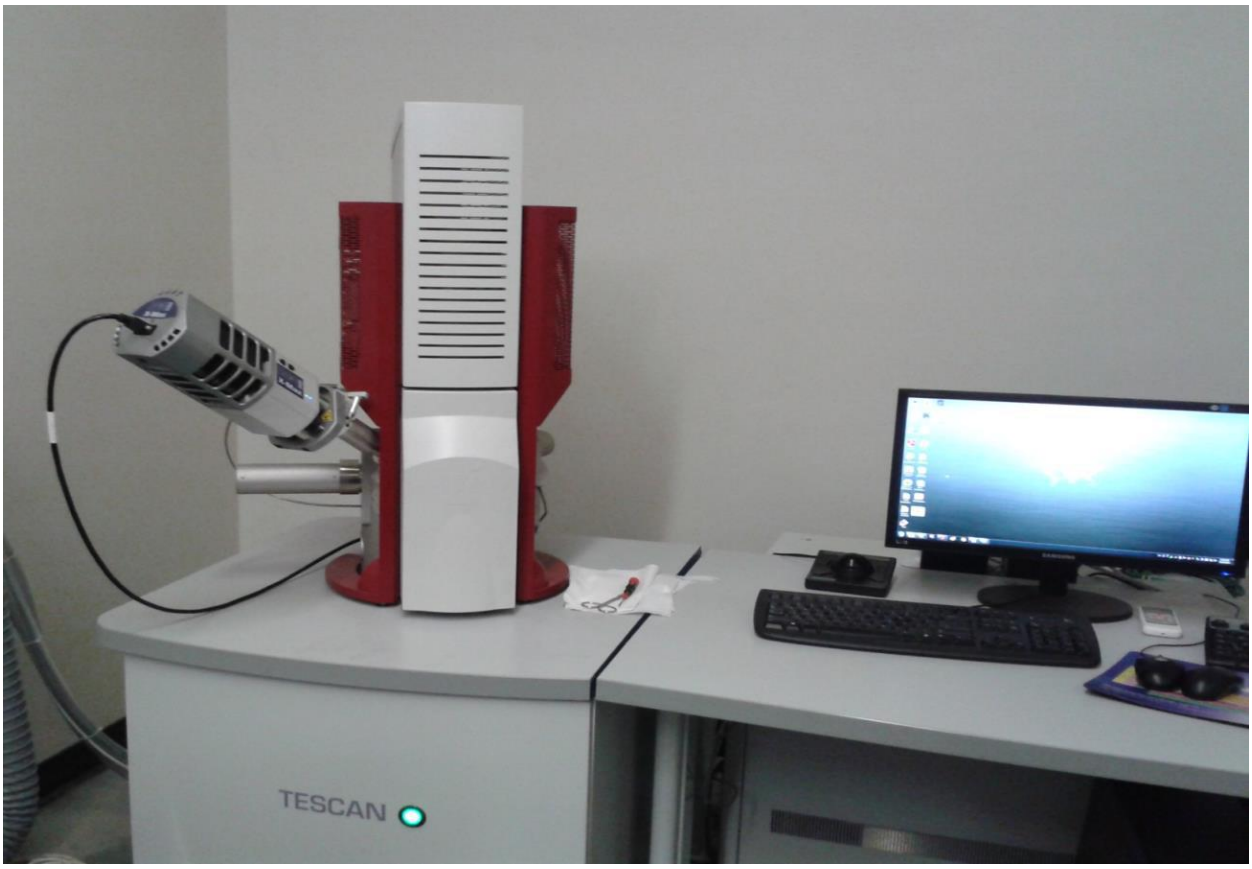


Figure 3:4: Photograph of Scanning Electron Microscope (MIRA3 FEG-SEM).

3.4.4 Viscosity Measurements

Many equipment were used in the literature to measure viscosity of fluids such as concentric cylinder, plate and cone methods. To measure viscosity of nanofluids Stormer viscometer was chosen which is shown in figure 3.5. This equipment can measure dynamic viscosity, it can be converted easily to kinematic viscosity. This type of viscometers was used to measure viscosity because still the nanoparticles phenomena still not clearly understood due to the opacity of the nanofluids. Viscosity will be measrured by measuring the time required to complete 100 revolution in the response of the selected weight.

Different weights could be used but for nanofluids at low concentrations it is recommended to use low weights because he increasing in viscosity is not significantly.

There is a simple procedure of doing the viscosity experiments. Measurements will be taken at a range of temperatures from 25 ℃ to 55 ℃ for all of the samples. Firstly sample will be heated to high temperature about 70 C, then the sample will be transferred to the main cup where the rotations takes place, in the main cup there is a thermometer holder to keep measuring temperature while taking viscosity measurements. While taking results, measurements are taken by cooling the samples by the effect of the ambient air only without forced cooling. This equipment is a well-known equipment for measuring high shear liquids like oils, the uncertainty of the measurements less than 1.5 % of its mean value for the entire range.

Figure 3:5: Photograph of Stormer viscometer

3.4.5 Differential Scanning Calorimeter (DSC)

Differential scanning calorimeter is an equipment used to measure changes in enthalpy such as heat flow and specific heat capacity as a function of temperature. Samples when they operated in the equipment, they exposed to a linear temperature which in turn will enable the equipment to measure the heat flow and specific heat capacity as a function of temperature.

In this work, differential scanning calorimetry, (DSC Q1000, Model TA Instruments, USA), connected with rapid cooling system, RCS 90 using Nitrogen gas, was used.

Nitrogen gas will be used as inert which used to cool down the flange temperature and the sample itself. The flow rate of the nitrogen must be fixed at 50 ml/min.

Procedure of this test includes weighting the sample to be between 12 – 15 mg within a hermetic aluminum pans, then closing the pan with special tool designated specially for this objective. After placing the pan in its place, operator can set his special procedure, for measuring specific heat capacity of nanofluids the procedure includes equilibrating the sample temperature to 10 °C, then stabilizing the temperature of the sample for 1 min after that the main step started by heating the sample by heating rate 3 °C/min from 10 °C to 80 °C. During heating step, in the main procedure, data storadge must be inserted within the procedure in order to get the data (specific heat capacity vs. temperature) in an excel sheet.

Specific heat capacity will be analyzed for all of samples to study the effect of surfactant concentration and concentration of CNT nanoparticles. Other observations could be noticed from specific heat capacity measurements especially from heat flow curve which can give a good indication about boiling and freezing temperatures which opens the door for other advantages of nanofluids. DSC equipment shown in figure 3.6.

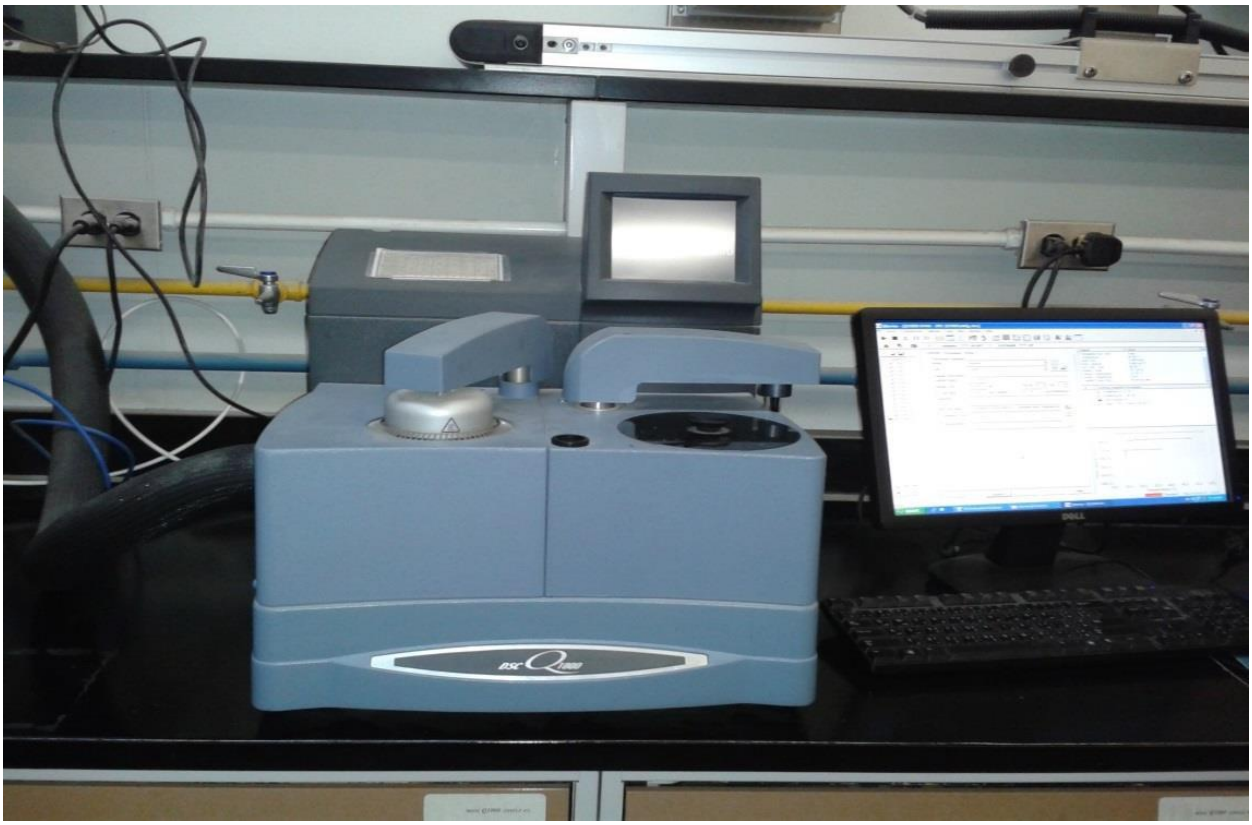


Figure 3:6: Photograph of DSC Q1000, Model TA Instruments.

3.4.6 Thermal Conductivity Analyzer

Thermal conductivity analyzer is an equipment used to measure the thermal conductivity of liquids, solids, powders, pastes and foams. This equipment “Mathis TCi” used interfacial, one side with constant and stable heat source to the sample and heat reflectance sensor that applies a momentary. In addition of thermal conductivity this equipment could measure effusivity of the sample.

The main sensor will apply a known current which provides a little amount of heat. This heat will make an increase in the temperature at the interface between the sample and the sensor around two degrees. This increase in the temperature make a voltage drop in the sensor. The rate of increasing in sensor voltage used to estimate the physical and thermal

properties of the sample. Material which highly insulative shows steeper in the voltage rise. TCA is shown in figure 3.7.

The procedure of performing tests including heating the samples using hot plate to a temperature about 60 C, then samples were put in a special 40 ml beaker and the main sensor of the equipment will be immersed in the solution, after that measurements appears on the screen of the software. Thermal conductivity and effusivity could be estimated by this equipment, measurements in this test was done for a temperature range 20 ℃ to 50 ℃. Thermal conductivity done in order to see if there is any effect of surfactant on thermal conductivity measurements.

Figure 3:7: Thermal conductivity analyzer.

3.4.7 Density Measurements

Density measurements were done using a simple procedure that include weighting a specific volume of sample and then calculating the ratio of mass to volume which is equal

to the density. Tests was done at room temperature and randomly results will be done at different temperatures to see if there is any significant effect of temperature. The selected volume was 10 ml in order to get high accurate results. It was expected that surfactants may affect density.

3.5 Implementation the CNT-water Nanofluid

After characterization of nanofluids, the best nanofluid conditions will be applied in the shell and tube heat exchanger to study the heat transfer enhancement of the nanofluid, also to study the pressure drop within the tube side. Properties of the shell and tube heat exchanger used and operating conditions are summarized in table 3.1, and the experimental set up of the heat exchanger are shown in figure 3.8. Monitoring flow rates of the heat exchanger will be done by a software that was connected to the equipment. This equipment manufactured by QVF Germany Company for heat exchangers, while heater and chiller manufactured by LAUDA company.

Temperatures for both chiller and thermostat are controlled by set up controller connected with each one.

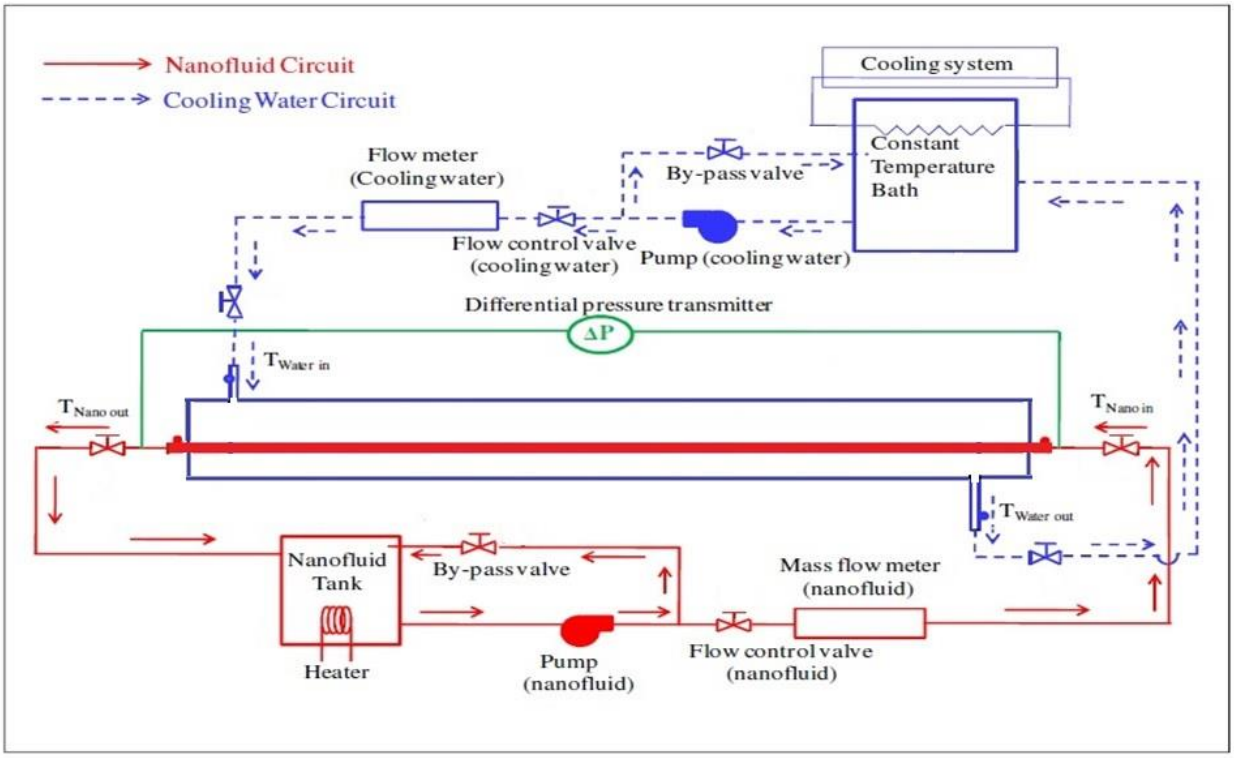


Figure 3:8: Experimental set up of heat exchanger that used in measurements

Table 3:1: Properties and operating conditions of the shell and tube heat exchanger

Type of the shell and tube heat exchanger	Concentric tube heat exchanger
Tube material	Stainless steel
Tube length	3 m
Inner diameter of the tube	10 mm
Thickness of tube	1 mm
Shell Material	Glass
Shell diameter	23.4 mm
Thermocouple type	K-type
Pressure drop measurements	Digital pressure transducers
Hot fluid	Nanofluid
Cold fluid	Distilled water
Concentrations of nanoparticles	0.1, 0.3 and 0.5 wt%
Hot fluid mass flow rate	100 – 600 kg/hr
Cold fluid mass flow rate	230 kg/hr
Set up temperature of the hot fluid	40 °C
Set up temperature of the cold fluid	25 °C

In order to minimize heat loss, two steps were done, the hot fluid which is the nanofluid will be in the tube side and also all of the heat exchanger was isolated using rock wool around the shell. Two water paths as shown in figure 3.8 for both of the hot and cold fluids. Four thermocouple of the mentioned type were connected at the inlet and outlet of the hot and cold fluids.

3.6 Heat Transfer Measurements of the Nanofluid

After applying the nanofluid in the heat exchanger, the flow rate will be changed from 100 – 600 kg/hr by increasing and decreasing the flow. Each reading will be repeated three times and the average reading will be considered. The heat transfer must reach to steady state conditions, in order to achieve that heat exchanger left to a period of time approximately 20 min. Then 4 temperatures and pressure drop will be recorded.

Equation 3.1 used to calculate the heat transfer rate while equation 3.2 used to calculate the overall heat transfer coefficient.

$$Q = \dot{m}_n \, Cp_n \left(T_{n,in} - T_{n,out} \right) \qquad \text{equ. 3.1}$$

$$U = \left(\frac{Q}{A \times \Delta T_{lm}} \right) \qquad \text{equ. 3.2}$$

To study the heat transfer enhancement, before each concentration, water will be applied instead of the nanofluid to study the amount of energy transferred by applying water then nanofluid will be applied to observe the difference in heat transfer between both of the fluids.

New tubes will be used for each concentration, that means after applying each concentration, heat exchanger will be cleaned and then the new cycle started by applying water and then the new concentration of the nanofluid. Specific heat capacity values which will be used in heat transfer calculations are the obtained values from DSC for water and nanofluid.

CHAPTER 4

RESULTS AND DISCUSSION

A series of experiments were performed to determine the stability, viscosity, heat capacity, thermal conductivity, heat transfer and pressure drop for various nanofluids.

4.1 Surface characterization of the raw and impregnated CNTs

Figure 4.1 displays the field emission scanning electron microscopy FE-SEM images of the raw carbon nanotubes at low and high magnifications. The diameters of the CNTs varied from 20–40 nm with an average diameter of 24 nm, while their length varied from 10–30 μm. Backscattering FE-SEM images for two different samples with CNTs doped with 1 and 10 wt% of Fe_2O_3 nanoparticles are shown in figure 4.2. These images confirms the presence of nanoparticle ions on the surfaces of the CNTs. At a low Fe_2O_3 nanoparticle concentration (1 wt%, Figure 4.2 (a)), the particles were widely distributed on the surfaces of the nanotubes in the form of very small crystalline particles with diameters varying between 1–5 nm. At high concentration (10 wt%, Figure 4.2 (b)), the particles agglomerated to form large crystalline nanoparticles with diameters varying from 1–15 nm.

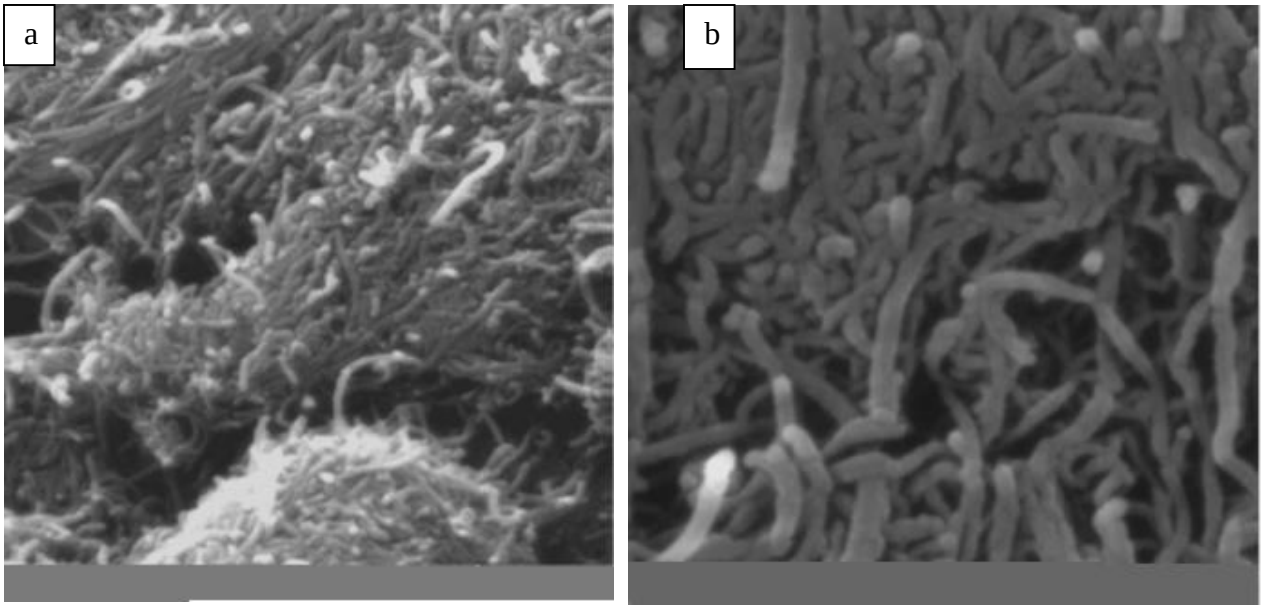


Figure 4:1: FE-SEM images of CNTs at (a) low magnification and (b) high magnification.

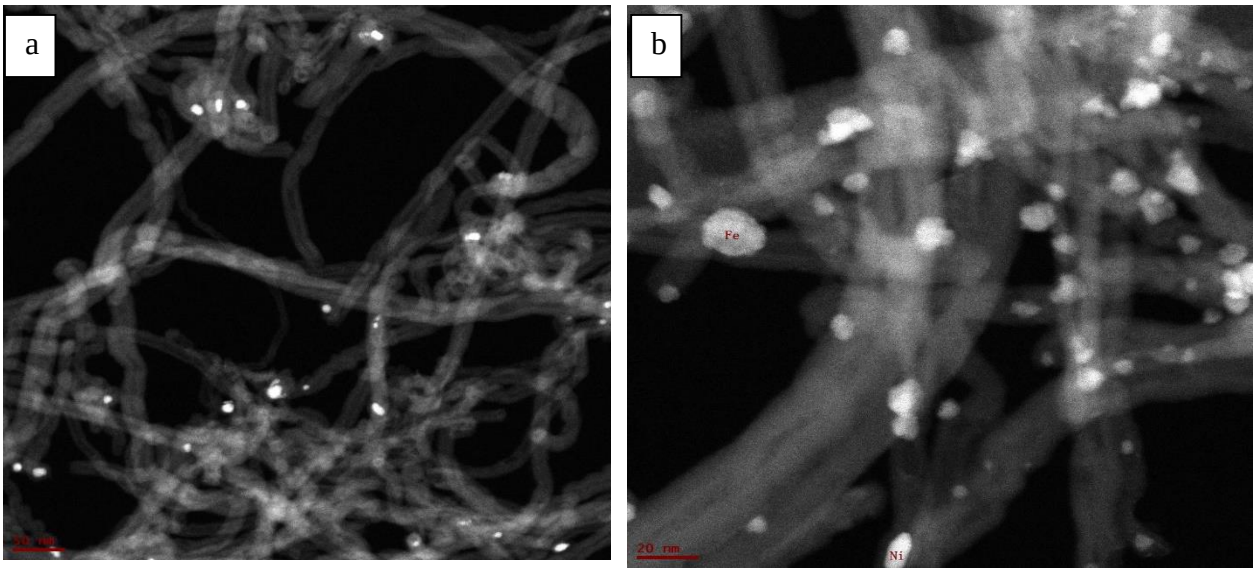


Figure 4:2: Backscattering FE-SEM images for CNTs doped with (a) 1 wt% and (b) 10 wt% of iron oxide nanoparticles.

Transmission electron microscopy FE-TEM was performed to characterize the structures and sizes of the nanotubes and iron oxide nanoparticles and to observe the configuration of the doping of the iron oxide nanoparticles on the surfaces of the CNTs are also observed by TEM. The HR-TEM image of the raw nanotubes is presented in figure 4.3 (a). This

image shows nanotubes that are hollow and tubular in shape with many deflection sites. Figure 4.3 (b) shows a typical HR-TEM image of Fe_2O_3-CNT. From this image, iron oxide nanoparticles can be observed on the nanotubes surface. It was found that the iron oxide nanoparticles covering the surfaces of the CNTs had an average particle size of ~6 nm, which is consistent with the backscatter FE-SEM results. It was also noted that the structure of the CNTs was not damaged during the impregnation process.

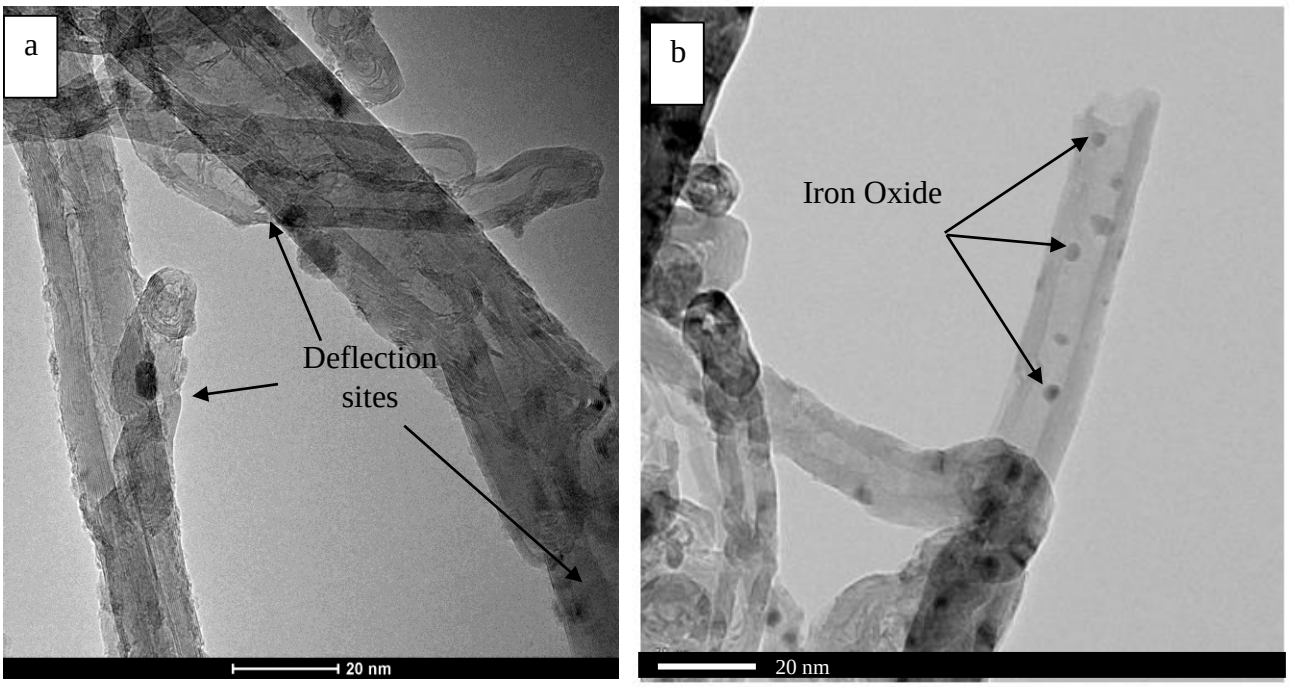


Figure 4:3: HR-TEM images of (a) CNTs and (b) Fe2O3-CNT.

To confirm the presence of the iron particles on the CNTs, Elementary diffraction X-ray (EDX) analysis has been conducted along with FE-SEM studies. Figure 4.4 shows the EDX spectrum of 1wt% and 10% of doped iron oxide on the surface of CNTs, that confirms the presence of iron oxide particles.

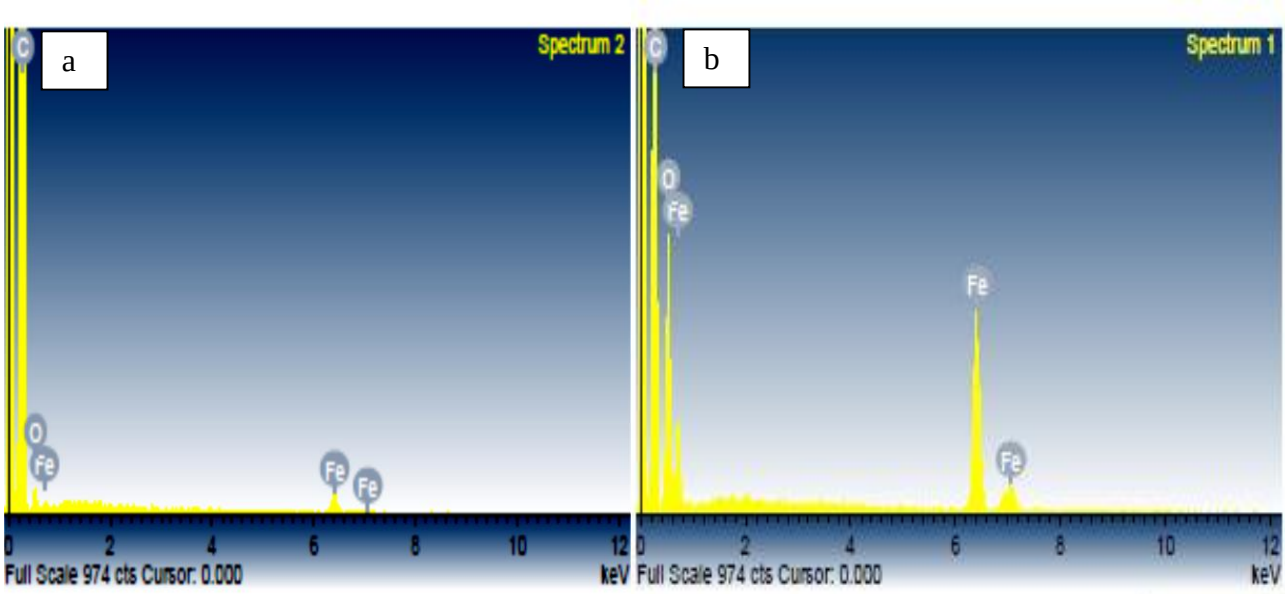


Figure 4:4: EDX analysis of CNTs impregnated with iron oxide (a) 1 wt% and (b) 10 wt%.

Table 4:1: EDS analysis of CNTs and Fe_2O_3-CNT

Element	CNT Sample		
	Raw CNTs	Fe_2O_3-CNT (1%)	Fe_2O_3-CNT (10%)
	Weight %	Weight %	Weight %
C	98.50	94.42	85.01
O	1.50	4.14	4.52
Fe	-	1.44	10.47
Total %	100	100	100

4.1.1 Thermal Degradation Analysis

The thermal oxidation study of materials is important as it determines the upper temperature limit for the use for a material. This can be studied using thermal gravimetric

analysis (TGA) and derivative thermogravimetric analysis (DTG). Figure 4.5 depicts the TGA and DTG results for the carbon nanotubes with and without iron nanoparticles. The analysis was performed in air with heating rate 10 °C/min. In the thermogram, the initial degradation of CNTs starts at approximately 550°C, reaches a maximum weight loss at approximately 600 °C and completes at approximately 670 °C, as revealed by the DTG curve. For CNTs impregnated with 1 wt% iron nanoparticles, the initial oxidation starts at 500 °C, reaches a maximum weight loss rate at approximately 550 °C and completes at approximately 600 °C, as shown in Figure 6 (b). When the loading of Fe_2O_3 was increased from 1 to 10 wt%, the oxidation peak in the thermogram shifts to lower temperatures, so that the oxidation starts at 450 °C with a maximum weight loss rate at 500 °C and completes at 540 °C. It can be observed that; the iron oxide particles doped on the CNTs surface serves as a heat accelerator for the heat transfer to the body of CNTs. It was observed that the iron oxide doped CNTs degrades faster than the undoped CNTs .

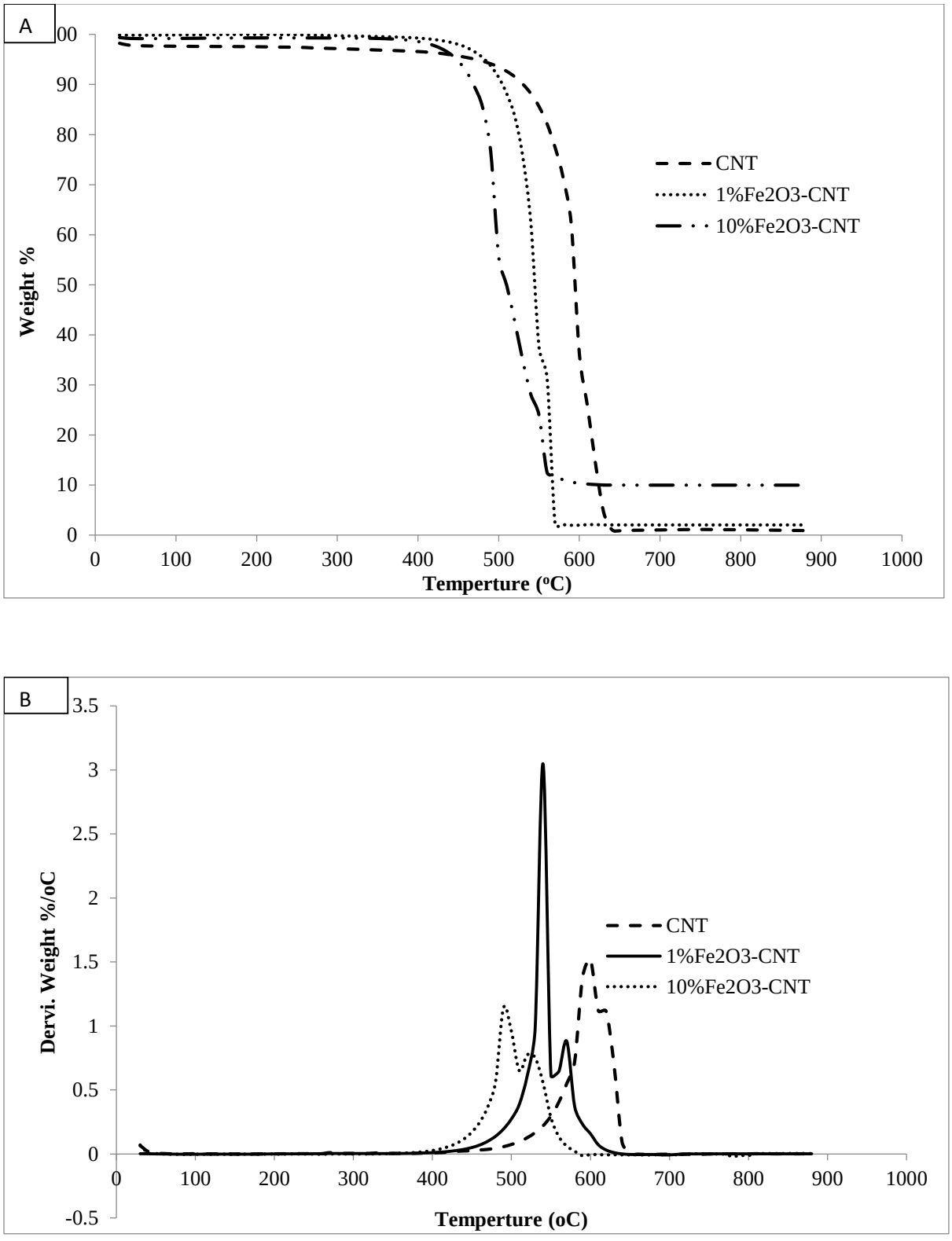


Figure 4:5: Thermogravimetric analysis (TGA) and (b) derivative thermogravimetric analysis (DTG) results for raw and doped CNTs

4.2 Stability and Physical Observation of Nanofluids (NFs)

Stability of the nanofluids is one of the key factors for application of the NFs, especially in heat transfer. There are two common methods for measuring the stability, dispensability and suspendability of the nanoparticles into the base fluid. The first method implies UV–vis spectrophotometry to measure quantitatively the colloidal stability of the dispersions [24]. The second method involving measuring the volume of nanoparticles sediment at different sediment time. In this work, the stability of the nanofluids visualized by checking the sedimentation of carbon nanotubes with different surfactant.

In this work four different weight % (0.1, 0.3, 0.5 and 1 wt %) of CNTs were dispersed into fixed volume of base fluid (water), using three different surfactants namely Poly Vinyl Pyrrolidone (PVP), Sodium Dodecyl Sulfate (SDS) and Gum Arabic (GA). Three different ratios of CNTs to Surfactants, i.e (1:0.2, 1:0.5 and 1:1) were used to find the optimum ratio for the total dispersion of CNTs without sedimentation. The carbon nanotubes and the surfactants were mixed in fixed volume of water followed by sonication for 30 min to break Van der Waals forces between the CNTs and to reduce the agglomeration and warp the individual CNT with the inonic surfactant. The prepared samples were kept for long time to observe the sedimentation of nanotubes. By visual observation of the nanofluid, the CNTs: Surfactants ratio of (1: 0.2) found to be unstable and most of the CNTs were precipitated after 36 hours (as figure 4.6). Increasing the ratio to 1:0.5 increases significantly the dispersion of CNTs and reduces the agglomeration. A little agglomeration of CNTs were observed after three months for sample with CNTs: SDS ratio of 1:0.5 ratio while a good dispersion and homogeneous solution were observed for both samples with

CNTs:GA and CNTs:PVP. After 6 months of the preparation of the nanofluid solutions, almost half of the CNTs were aggregated using SDS while for other two surfactants (GA and PVP), the samples were stable. Further increase in the ratio of CNTs to Surfactants to 1:1 formed a stable, well dispersed homogeneous solutions for more than 6 months, for all the surfactants at different concentrations of CNTs (As shown in Figure 4.7). Based on these observations, the ratios of CNTs to Surfactants (1:0.5 and 1: 1) were found to be the optimum and were chosen for all the remaining experiments. Stirring and heating were also applied to the sample with CNTs: Surfactants (1:0.5 and 1: 1), to study the effect of fluid movement and high temperature, on the stability of the nanofluids. It has been found that; the fluid was stable at high temperature and stirring and precipitation was observed for the CNTs.

Figure 4:6: CNT-Water nanofluid using 1:0.2 GA after 36 hours for different CNT loading.

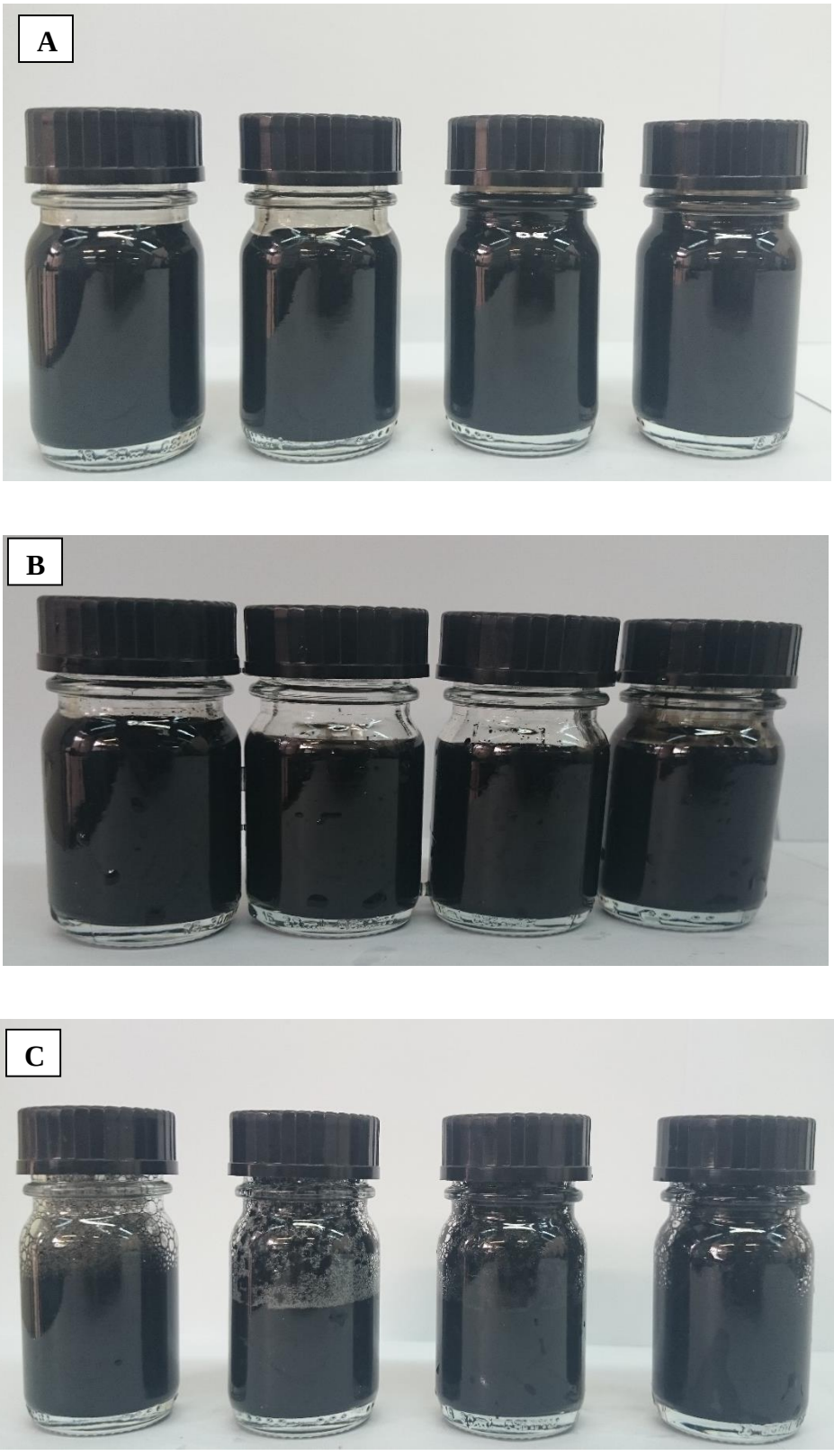


Figure 4:7: (A, B, C) CNT-Water nanofluid using 1:1 GA, PVP and SDS after 6 months

4.2 Effect of Surfactants on Thermal Conductivity of the Raw CNTs Nanofluids

As shown in Table 1:1, the thermal conductivity of CNTs is 2000 times higher than the thermal conductivity of water. To attain the maximum enhancement of the thermal conductivity of the nanofluids, the nanoparticles have to be well dispersed in the base liquid. Large particles are easy to agglomerate and precipitate out from the base fluids, which lead to a reduction in the thermal conductivity. This is due to formation of the sediment layer at the surface of the tube that increase the thermal resistance and reduce the heat transfer capacity of the fluids.

In this work the CNTs were well dispersed and homogenous solutions of CNTs to Surfactants ratio of (1:0.5) with water as base fluid were formed. The thermal conductivity of the prepared solutions at different concentrations of CNTs (0.1, 0.3, 0.5 and 1wt %) with different surfactants (GA, SDS and PVP) were measured over the temperature range of 20 ^{o}C to 45 ^{o}C using TCI technique. As seen in figure 4.8, the nanofluid thermal conductivity increases from 5 to 10%, with increase in temperature from 20 ^{o}C to 45 ^{o}C for the CNT with 0.1 wt% loading. The thermal conductivity was also observed to increase with the increase in CNTs concentration. From the experimental results, it was concluded that CNT–water suspensions have noticeably higher thermal conductivities than the water base fluid. However, it was observed that, there is no effect of the surfactants type on the thermal conductivity of the nanofluids. The measured values of the thermal conductivity of the nanofluids with different types of surfactants (GA, SDS, and PVP) at different concentrations of CNTs were almost the same.

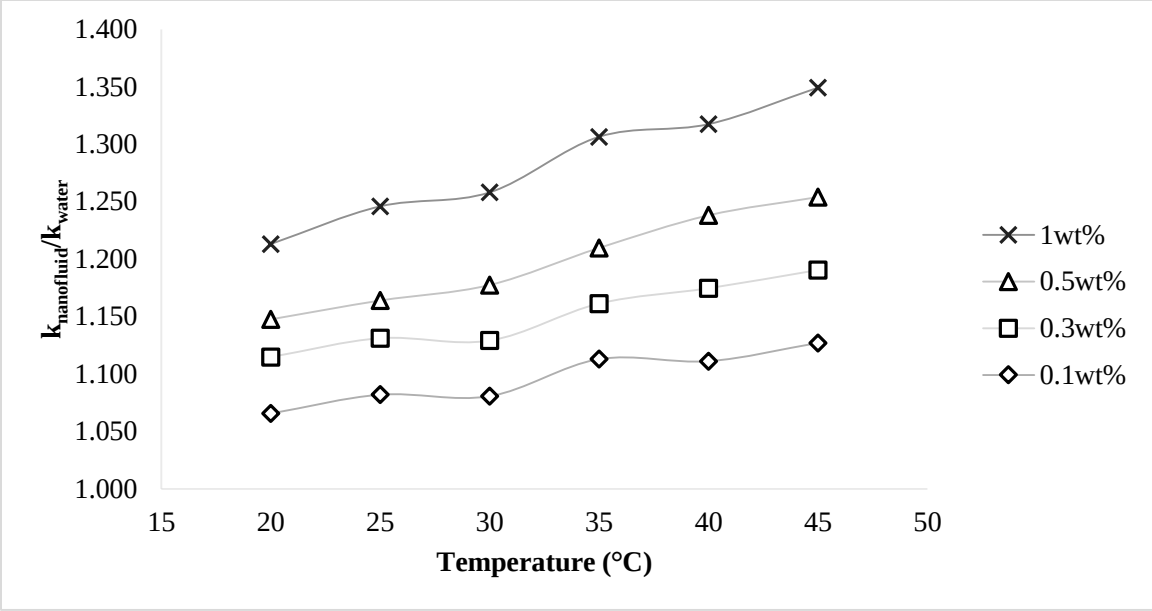


Figure 4:8: Effect of CNT concentration and temperature on the thermal conductivity

4.3 Effect of Surfactants on the Viscosity of Raw CNTs Nanofluids

Both viscosity and thermal conductivity of the nanofluids could be strongly associated with the microstructure and dynamics of the fluids. A commercial viscometer (Brookfield DV-I Prime) was used to measure the viscosity of the nanofluids over a temperature range of 20 ^{0}C to 55 ^{0}C. The measured viscosity of the base fluid (water) was found to be 1 cP which is in agreement very well with the literature value. Figure 4.9 shows the viscosity of the nanofluids as a function of temperature at different loadings of CNTs (0.1, 0.3 and 0.5 wt %) with three types of surfactants (GA, SDS and PVP) at 1:0.5 CNT: Surfactant ratio. For all nanofluids, regardless of the types of surfactants, the viscosity has increased with an increase in the concentration of CNTs. It can be observed that , GA and PVP has almost similar trends and values for the increment on the viscosity of the nanofluid at different CNTs concentrations with maximum increment of 40 % at higher CNTs loading of 0.5 wt % and 35 ^{0}C. While using SDS as surfactant, the viscosity increased up to 500 % at higher

CNTs loading of 0.5 wt % and 35 ^{0}C. This large increase in the viscosity of nanofluid using SDS as surfactants might cause real problem for heat transfer application because the viscosity is proportional to the pressure of the system and heat transfer. Figure 4.10 shows viscosity measurements for ratio 1:1 at two concentrations 0.1 and 0.5 wt%. At low concentration there is no effect of surfactant because of the modest concentration of GA and PVP. At 0.5wt%, viscosity was higher than 0.1wt%.

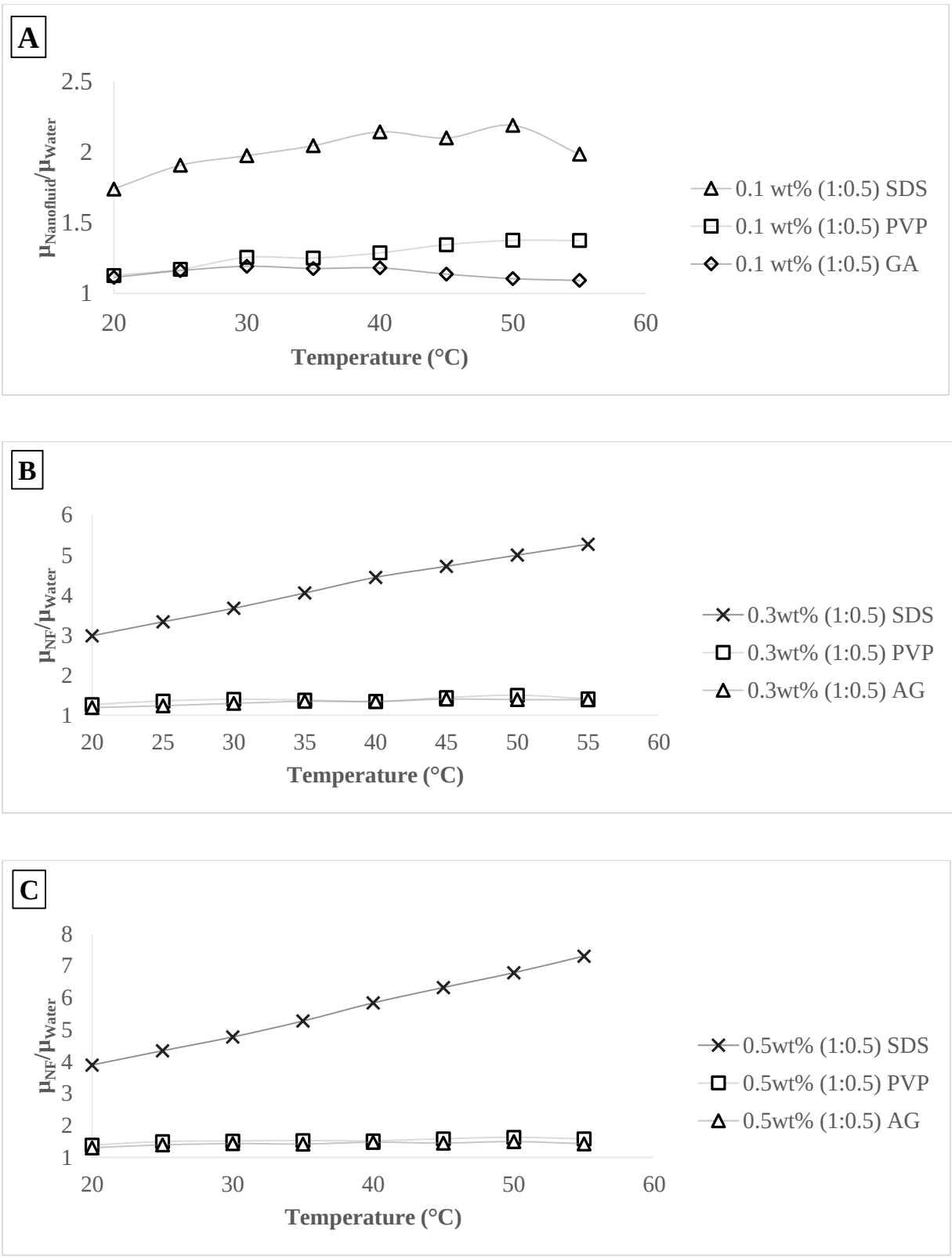


Figure 4:9: (A, B, C): Viscosity of (CNT-water) nanofluids as a function of temperature and CNT concentration for ratio 1:0.5

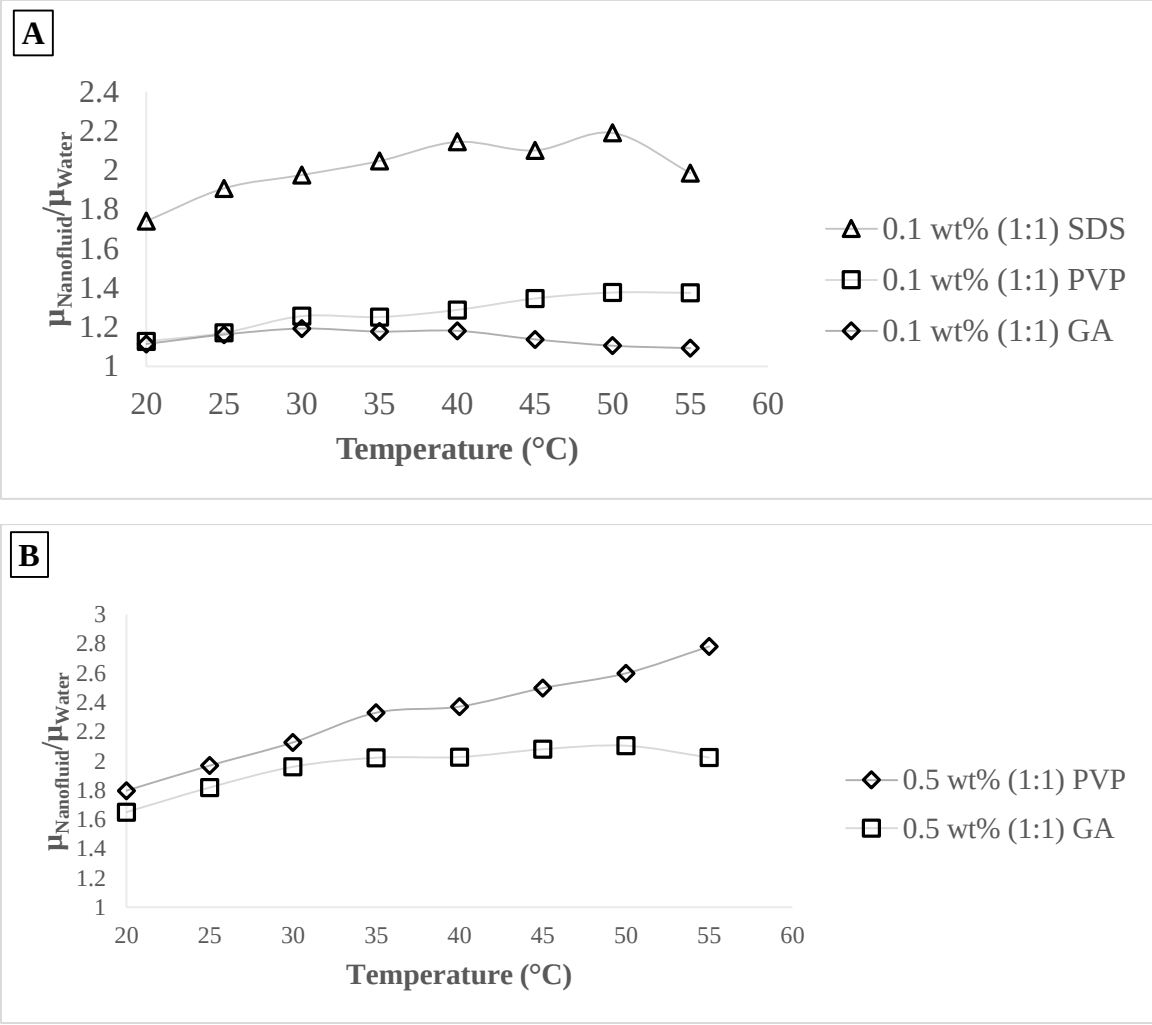


Figure 4:10: (A, B): Viscosity of (CNT-water) nanofluids as a function of temperature and CNT concentration for ratio 1:1

4.4 Effect of Surfactants on Density of the Raw CNT Nanofluids

Density is a very important property of a fluid that has major effect on the friction factor, pump loss and the Reynolds number. Figure 4.10 shows the measured values of the density at different weight fractions of CNTs with different ratios of surfactants. No major changes on the values of the density for different nanofluids solutions at different CNTs loading and different ratio of surfactants were noticed. It is almost constant values with 0.4 %

differences only. Adding small concentrations of these nanoparticles will not affect the major density of the base fluid, because the CNT has a closer density to water 1.2 gm/cm^3.

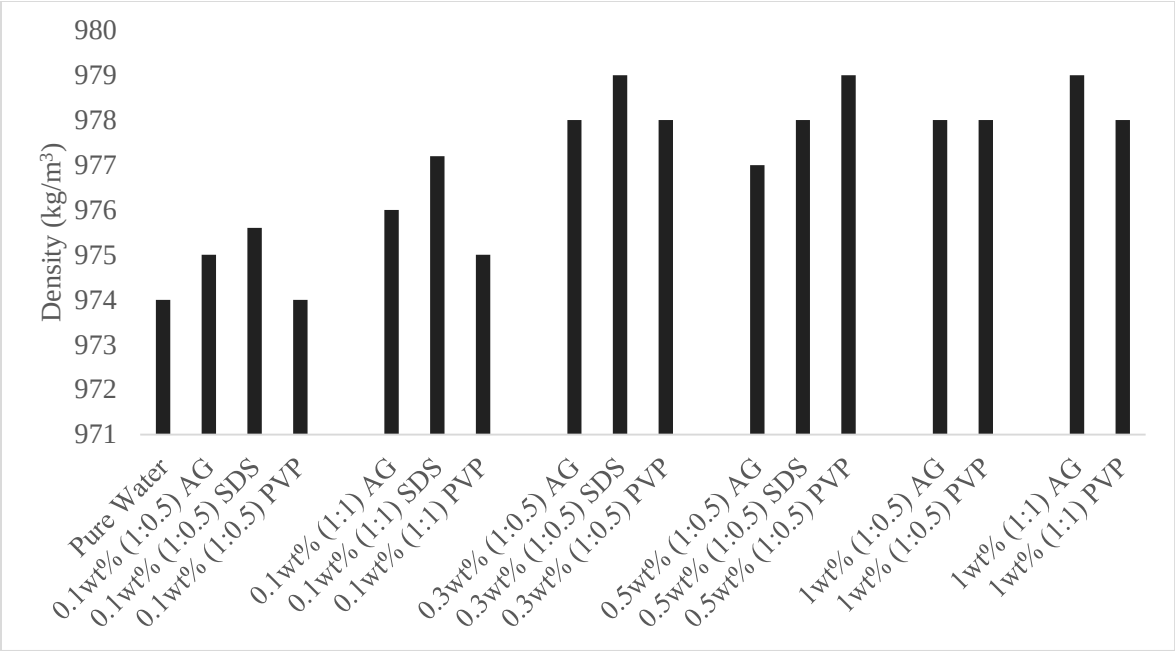


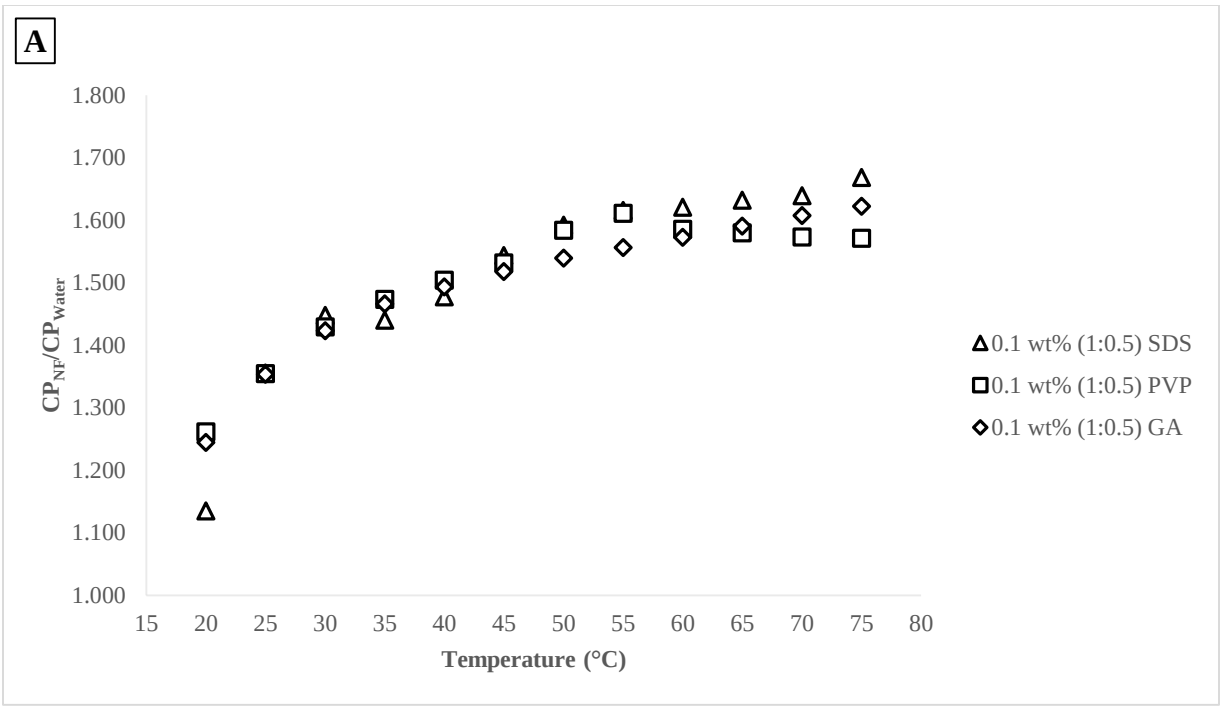
Figure 4:11: Density measurements of the CNT-water nanofluids.

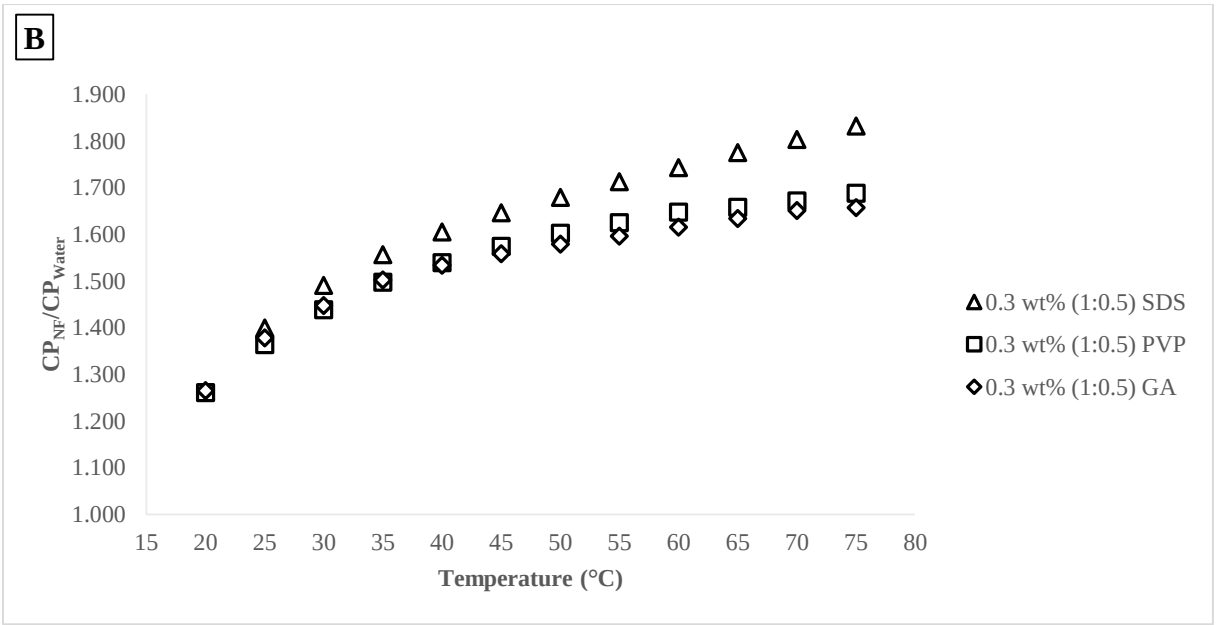
4.5 Effect of Surfactants on the Specific Heat Capacity of Raw CNT Nanofluids

In addition to thermal conductivity, density and viscosity, heat capacity is also an important parameter in determining the thermal performance of heat transfer fluids. In this work, the heat capacity of the nanofluids was measured using DSC (model TA-Q100). DSC measurements were performed from 20 ^{0}C to 75^{0}C with heating rate is 10 °C/min.

The heat capacity of water mixed with different concentrations of CNTs (0.1 to 1 wt%) and fixed ratio of surfactants (1:0.5) was investigated. Figure 4.11 shows the

enhancement of the heat capacity of the nanofluids (Cp_{nf}/Cp_w) as a function of temperature, where Cp_{nf} is the specific heat capacity of the nanofluid and Cp_w is the specific heat capacity of water. The results show that the heat capacity of the nanofluids increases significantly with increase in the concentration of CNTs. The maximum enhancements of the specific heat capacity of CNTs with surfactant raio of (1:0.5 GA, PVP and SDS) at a weight concentration of 0.1 wt% and temperature 35 °C were 45%, 45% and 35%, respectively. While increasing the loading of CNTs to 0.3wt % and 0.5 wt% under the same conditions has increased the heat capacities of the nanofluids up to 50%, 50% and 55% for 0.3 wt% and 50%, 50 % and 55 % for 0.5 wt %. It was noted that, no major effects of the CNTs loading on the heat capacity of the nanofluids above 0.1 wt% was observed.


A
1.800
1.700
1.600
1.500
1.400
1.300
1.200
1.100
1.000
CP_{NF}/CP_{Water}
15 20 25 30 35 40 45 50 55 60 65 70 75 80
Temperature (°C)
0.1 wt% (1:0.5) SDS
0.1 wt% (1:0.5) PVP
0.1 wt% (1:0.5) GA


B
1.900
1.800
1.700
1.600
1.500
1.400
1.300
1.200
1.100
1.000
CP_{NF}/CP_{Water}
15 20 25 30 35 40 45 50 55 60 65 70 75 80
Temperature (°C)
0.3 wt% (1:0.5) SDS
0.3 wt% (1:0.5) PVP
0.3 wt% (1:0.5) GA

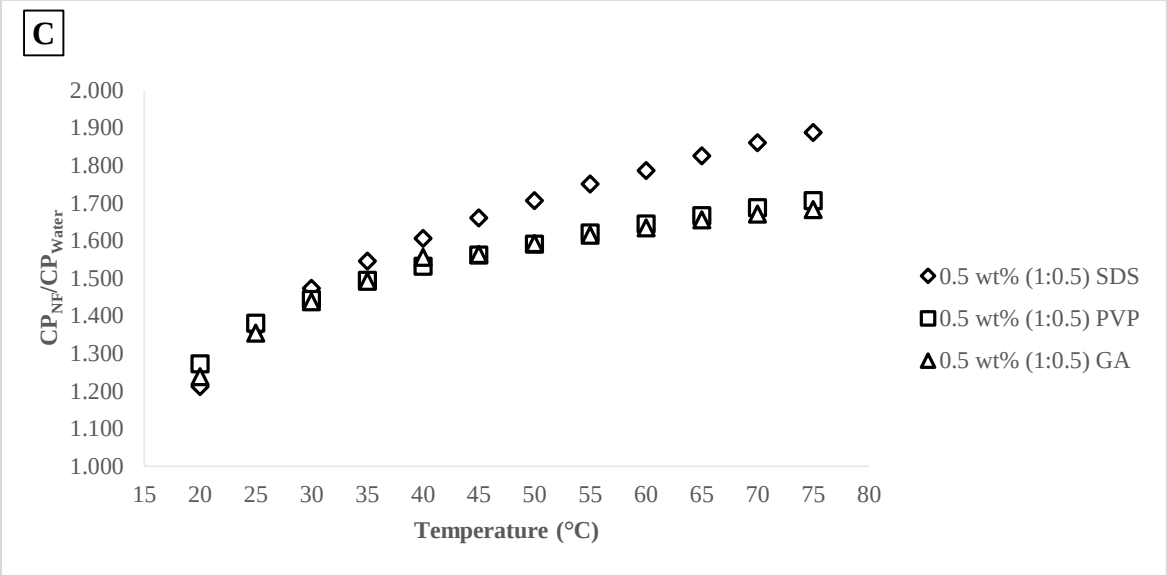


Figure 4:12: (A, B, C): Specific heat capacity enhancement for 0.1, 0.3 and 0.5 wt% CNT-water nanofluids with different surfatants

4.6 Application of the Raw CNT Nanofluids in Shell and Tube Heat Exchanger

4.6.1 Heat Transfer Rate Enhancement

A shell and tube heat exchanger was used to measure the heat transfer of the nanofluid. The measurement was taken under steady-state conditions. The inlet temperature of the nanofluids was fixed by a controlled heating bath at 40 °C, while their flow rate was regulated by a digital mass flow controller between 100–600 kg/h to maintain the nanofluid in the turbulent regime. Figure 4.12 shows the effect of different weight concentrations of CNTs (0.1, 0.3 and 0.5 wt%) with 1:0.5 ratio of GA on the enhancement of the heat transfer at different flow rates. It is evident that, the presence of CNTs can significantly increase the heat transfer rate. At Rynolds = 15, 000, the heat transfer of the nanofluid has increased up to 50% , 55% and 57 % at 0.1 wt % , 0.3 wt % and 0.5 wt%

of CNTs loading, respectively. Further increase in the flowrates of the nanofluid increases the heat transfer with maximum enhancement of 65% at 20,000 Re number and 0.5 wt CNTs loading. This large enhancement in the heat transfer of nanofluid could be justified by many reasons. For example, significant enhancement in the heat capacity and thermal conductivity of the nanofluid. The maximum enhancement of the thermal conductivity and the specific heat of the nanofuild at 0.5 wt% at temperature of 35 ^{0}C is 35 % and 40 %, respectively. It can be noticed that these two factors (thermal conductivity and the heat capacity) are not only the factors that affect the enhancement of the heat transfer of the nanofluids. The second reason might be due to the influence of Brownian motion. While Brownian movement of particles has a molecular timescale, it is manifested through particles that have inertia and respond to much higher timescales. At these higher timescales, the movement of the particles locally agitates the fluid, causing the increased heat transfer. Finally, the surface phenomenon might be responsible for enhancing heat transfer, where a small layer of CNTs coats the wall of the steel tube and enhances the thermal conductivity of the steel. However, further detailed study is needed to draw significant conclusions of the effect of these underlying mechanisms.

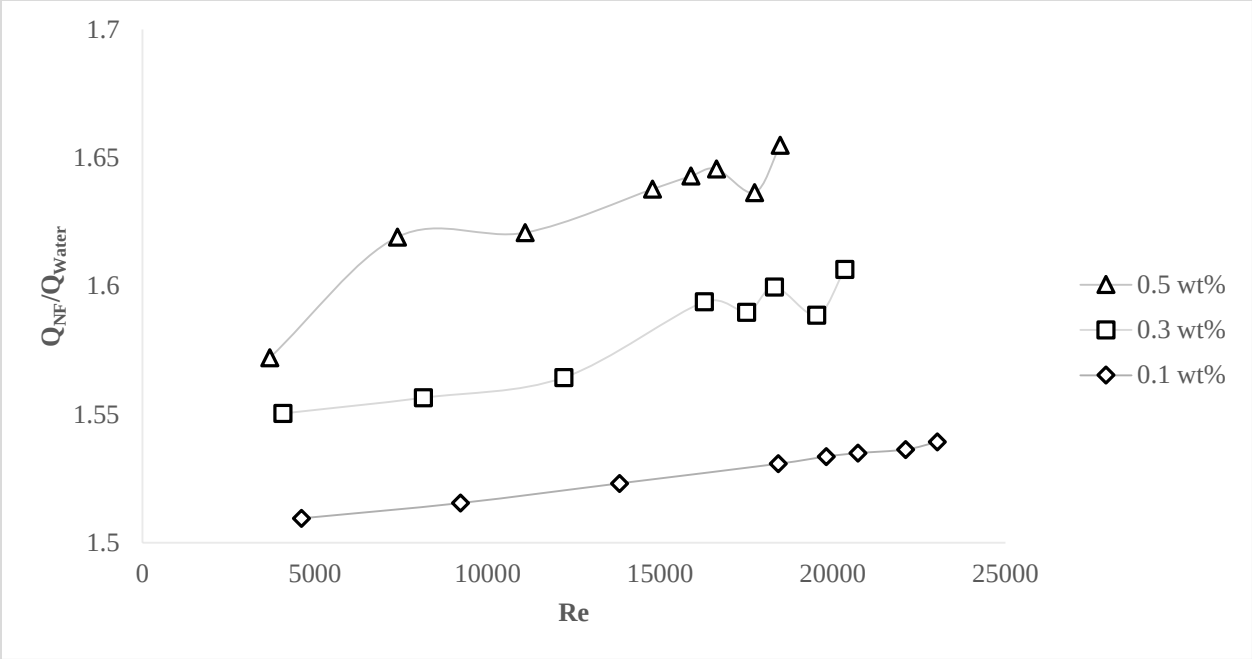


Figure 4:13: Heat transfer enhancement as a function of Reynolds number for different CNT loading

4.6.2 Effect of the Nanofluids on the Pressure Drop

A differential pressure transmitter was used to measure the pressure drop between the inlet and outlet tubes. The pressure drop was measured for the turbulent flow regime with mass flow rate varying from 100 to 600 kg/hr. Figure 4.13 shows the variation of the pressure drop as a function of the mass flow rate for different loading of CNTs (0.1, 0.3 and 0.5 wt%) with 1:0.5 ratio of GA nanofluids. No change in the pressure drop was observed with change in the concentration of CNTs in water. While it is well-known that the presence of nanoparticles increases the pressure drop of the system, in this work, the nanomaterials were not observed to affect the pressure drop of the system, owing to the very low surface roughness of the nanomaterials.

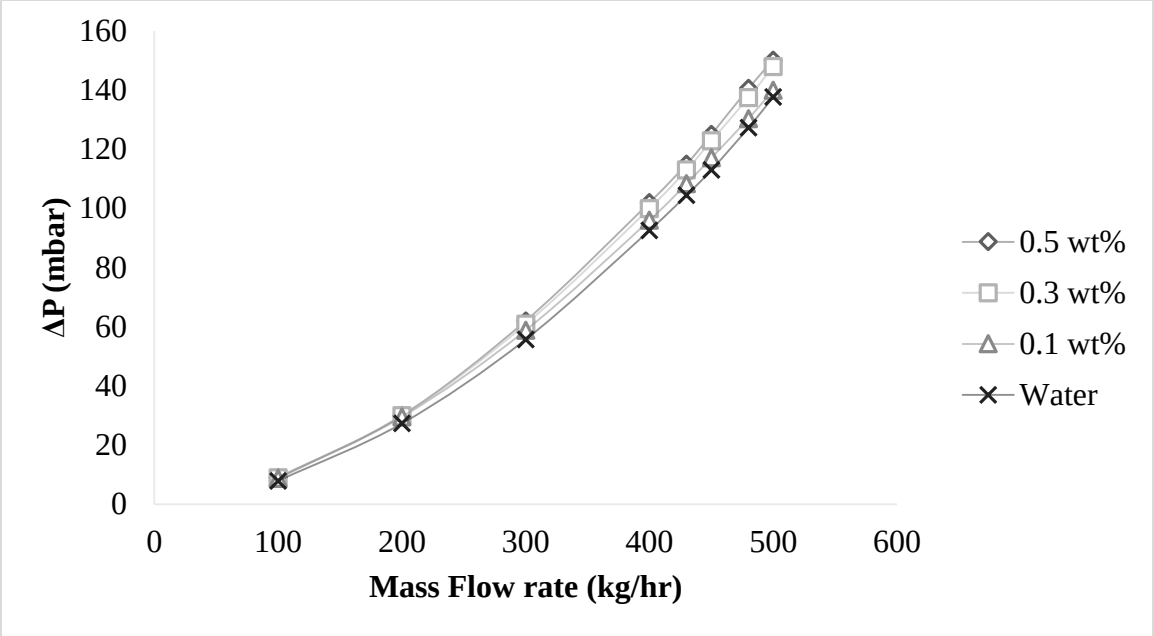


Figure 4:14: Pressure drop of CNT-water nanofluids using GA

4.7 Thermo-Physical Properties for Impregnated CNT Nanofluids with Surfactants

This section presents the results and discussions of the research finding of the impregnated CNTs with iron nanoparticles and their effects on the heat transfer of the nanofluid under different types of surfactant. The results and discussions have been divided into four sections. The first part discussed the effect of surfactants on thermal conductivity of impregnated CNT Nanofluids, while the second part discussed the effect of surfactants on viscosity of Nanofluids. The third and fourth parts discussed the density and the heat capacity of the nanofluid.

4.7.1 Preparation of Impregnated CNT Nanofluids

The traditional impregnation procedure was used to impregnate the surface of CNTs with different loadings of iron oxide (1wt% and 10wt %). As shown in previous part the ratio (1:0.5) surfactant was enough to disperse the CNTs into the nanofluid and therefore this ratio were fixed for I-CNTs nanofluid as shown in table 4.2

Table 4:2: Summary of prepared samples for impregnated CNT-water nanofluids.

Fe_2O_3 Loading	GA (1:0.5)		PVP (1:0.5)		SDS (1:0.5)	
1 wt%	0.1 wt%	0.3 wt%	0.1 wt%	0.3 wt%	0.1 wt%	0.3 wt%
	0.5 wt%	1 wt%	0.5 wt%	1 wt%	0.5 wt%	1 wt%
10 wt%	0.1 wt%	0.3 wt%	0.1 wt%	0.3 wt%	0.1 wt%	0.3 wt%
	0.5 wt%	1 wt%	0.5 wt%	1 wt%	0.5 wt%	1 wt%

4.7.2 Effect of Surfactants on Thermal Conductivity of Impregnated CNT Nanofluids

The thermal conductivity of impregnated CNT-water nanofluids with different loadings of iron oxide (1% and 10%) using (1:0.5) ratio of surfactants was measured using TCi thermal conductivity meter over a range of temperature from 20 °C to 50 °C. As shown in figure 4.15 the addition of iron oxide nanoparticles on the surface of CNTs does not affect the thermal conductivity of the nanofluids. It is almost similar phenomena, trends and results were observed for both non impregnated and impregnated CNTs.

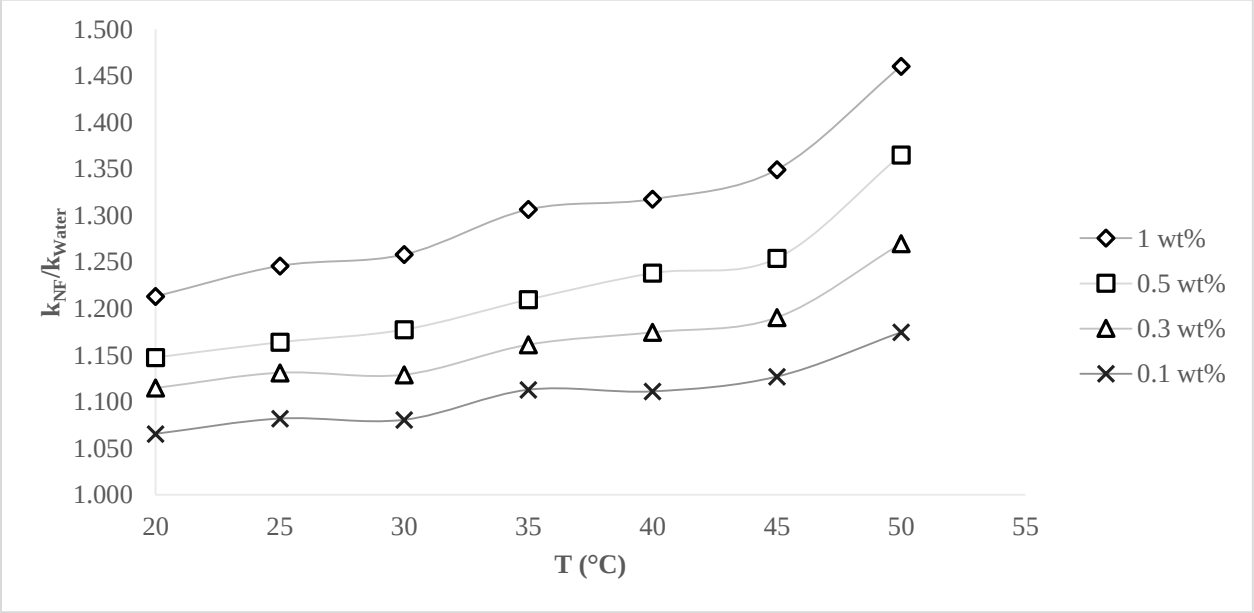


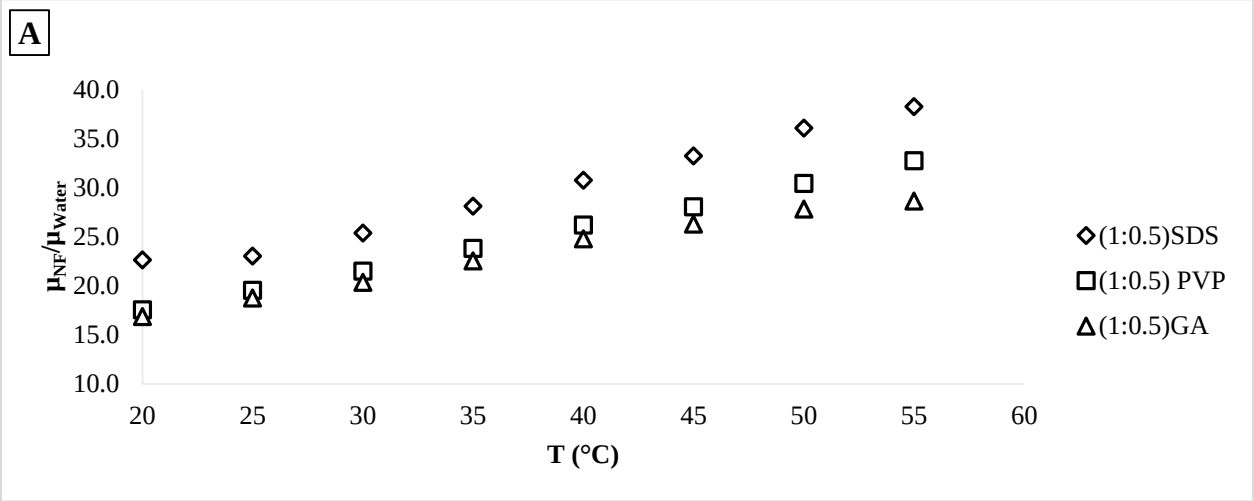
Figure 4:15: Thermal conductivity enhancement for different CNT concentration

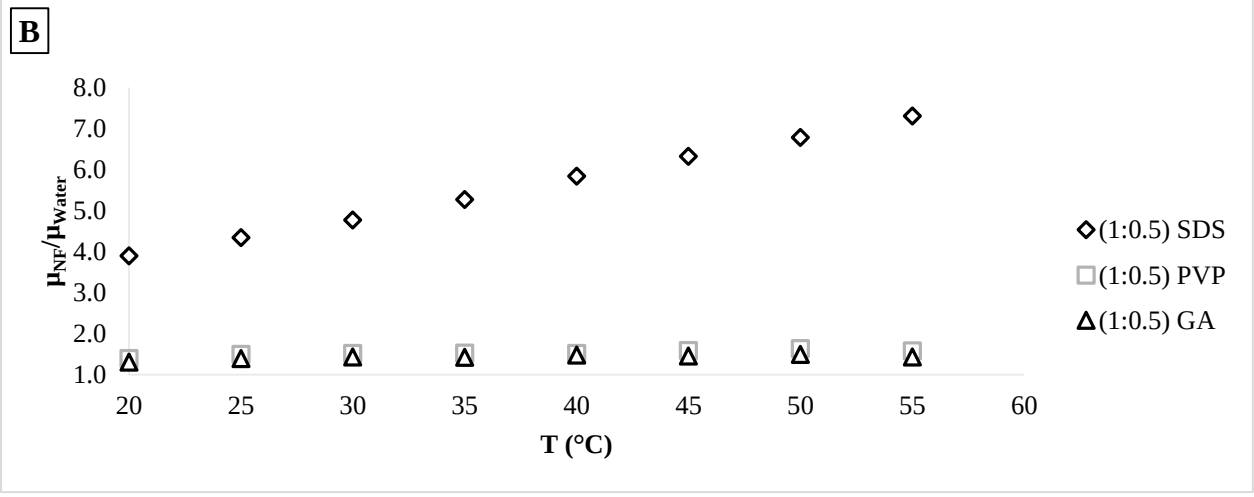
4.7.3 Effect of Surfactants on Viscosity of Impregnated CNT Nanofluids

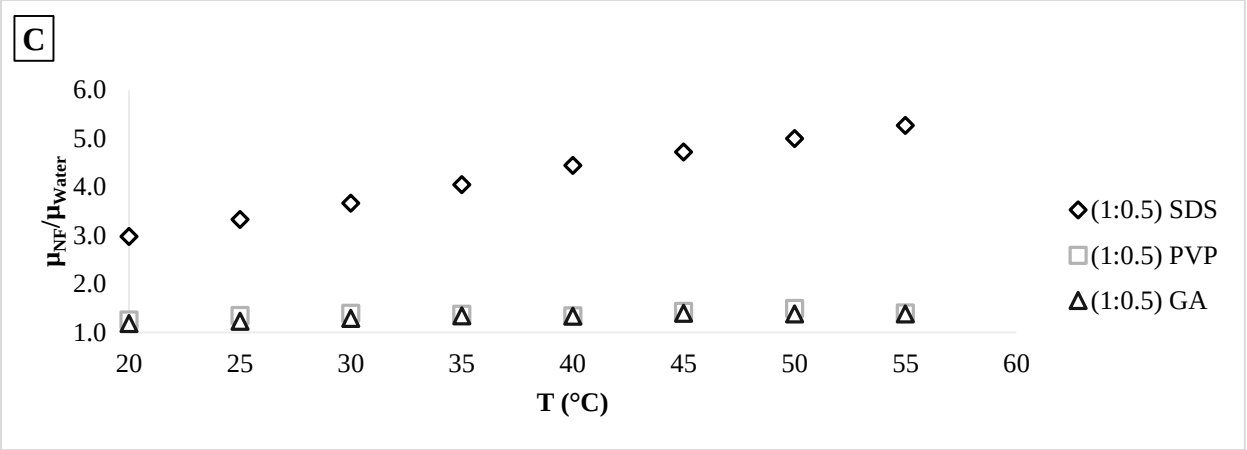
In this part, the viscosity of the impregnated CNT-water nanofluids was investigated under the same ratio of surfactant 1:0.5 and iron loading of 1 and 10 wt%. It was observed that there is no effect of the iron oxide on the viscosity of nanofluids; similar results were obtained at fixed concentration of CNTs for both 1% and 10% iron oxide. In addition to that; almost similar trends and results were obtained for both non impregnated and impregnated CNTs. The results indicate that the viscosity of nanofluids significantly increases with increasing in the concentration of the CNTs. However, the viscosity decreases as the temperature of the fluid increases. The microscopic view of the molecular level of the fluids can be used to determine the reasons which lead to the decrease on the viscosity of the fluid with the increase in temperature. The viscosity of the fluid depends on two major factors. The first factor is strength of the atomic bonding between the molecules which depends on the structure, shape and the size of the molecules. The second factor is kinetic energy of the molecules which direct proportion to the temperature of the system. It can conclude that, increase the temperature of the fluid will weaken the atomic bonding between the molecules and increase their kinetic energy which will lead to decrease the viscosity.

Figures 4.16 show the viscosity of nanofluids as a function of temperature at different loading of doped CNT with iron oxide for 1 and 10wt.%. The results showed that the viscosity of the nanofluid at different loading of CNTs doped CNTs at 0.1 wt. %, 0.3 wt. % and 0.5 wt. % with 1:0.5 GA and 35 ºC increases the viscosity of the nanofluid up to 17 %, 34 % and 41 % respectively. As shown in figure 4.16 there is no effect of the iron oxide

nanoparticles doped on CNTs on the viscosity of the fluid, since the viscosity of the fluid is function of the temperature of the fluid and the concentrations of the particles.







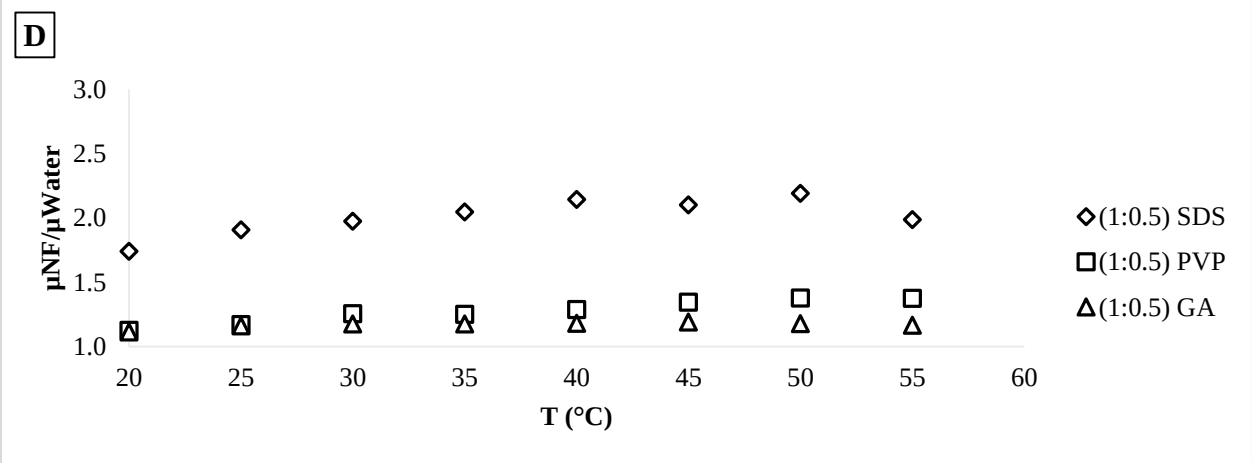


Figure 4:16: (A, B, C, D): Viscosity of nanofluids as a function of temperature at different loading of doped CNT with iron oxide for 1 and 10wt. % and different CNT loading 1, 0.5, 0.3 and 0.1 wt%.

4.7.4 Effect of Surfactants on Density of Impregnated CNT Nanofluids

The effect of I-CNTs on the density of nanofluid was investigated. No effect of the I-CNTs on the density of nanofluids. Figure 4.17 show density of nanofluids of the three types of surfactants for both 1% and 10% iron oxide nanoparticles. No major changes on the values of the density for different nanofluids solutions at different CNTs loading and different ratio of surfactants were noticed, it is almost constant values with 0.4 % differences only.

This can be illustrated that, the CNTs has almost closer density to water base which is 1.2 gm/cm^3, therefore adding small concentrations of these nanoparticles will not affect the major density of the base fluid.

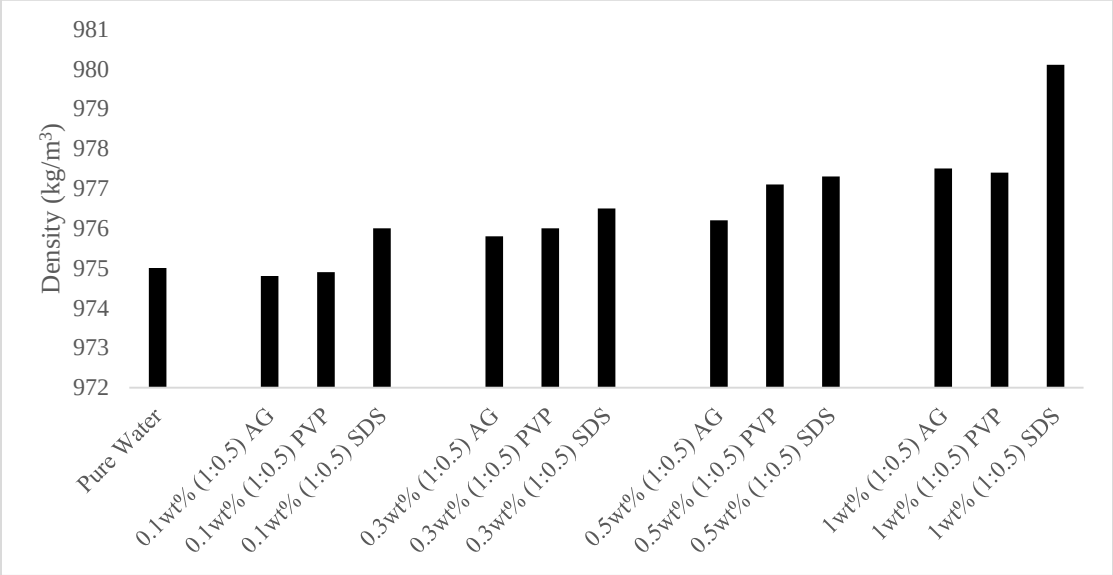


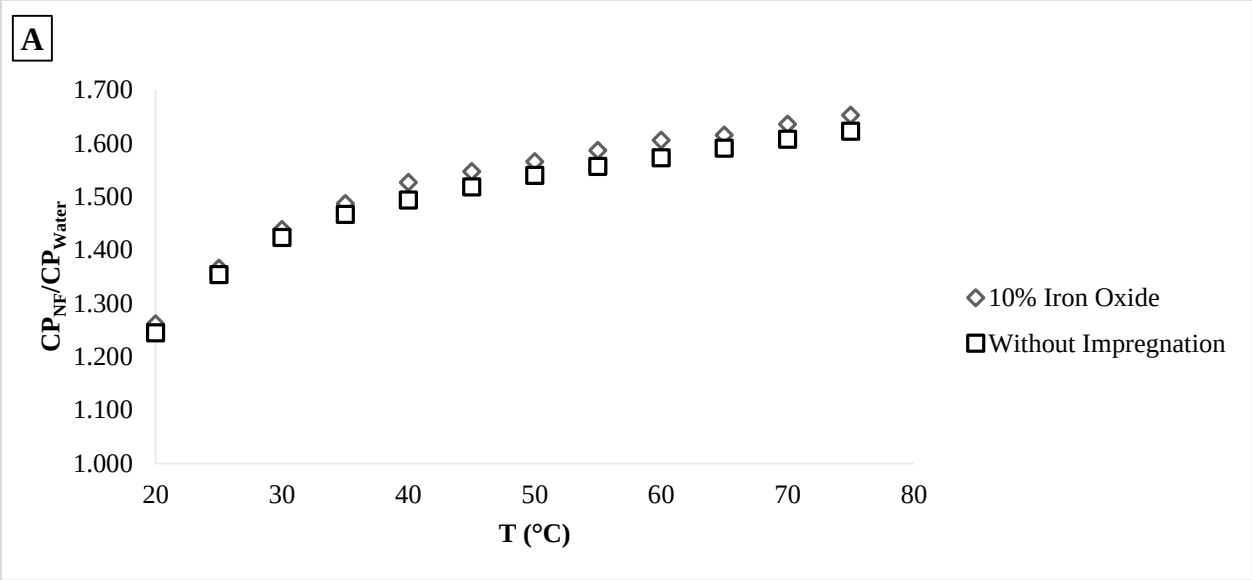
Figure 4:17: Density measurements of impregnated CNT with 1% and 10% iron oxide

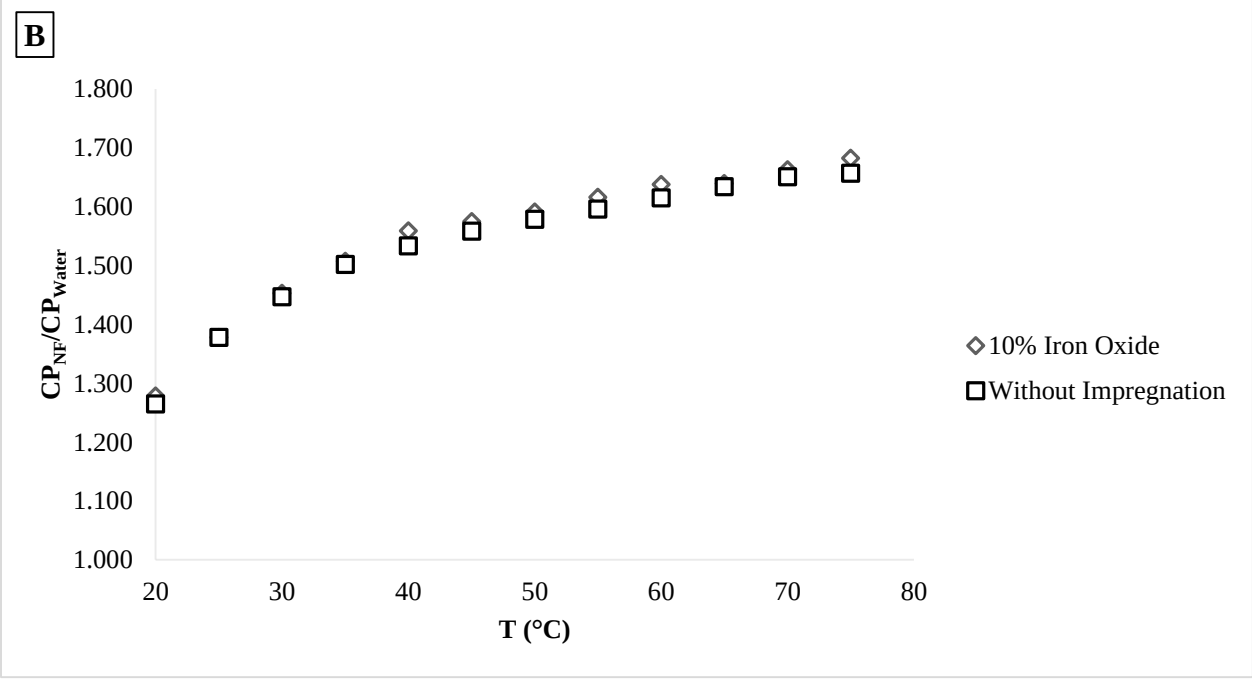
4.7.5 Effect of Surfactants on Specific Heat Capacity of Impregnated CNT Nanofluids

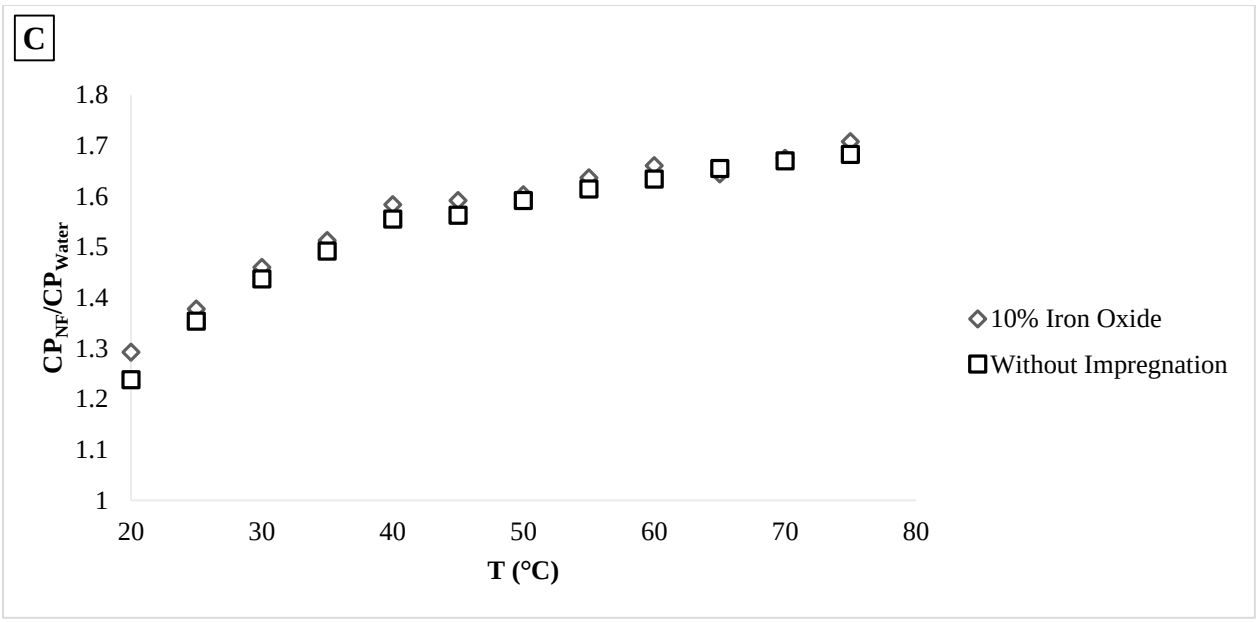
The heat capacity of water as base fluid mixed with different concentration (0.1, 0.3, 0.5 and 1wt.%) of I-CNTs at (1 and 10% wt.%) were investigated using DSC technique over a range of temperature from 25^{o}C to 50^{o}C at heating rate of 1.5^{o}C/min.

Figures 4.18 shows the enhancement of the heat capacity of nanofluids ($Cp,_{nf}/Cp_w$) as a function of temperature. Where $Cp,_{nf}$ is the specific heat capacity of nanofluids and Cp_w is the specific heat capacity of water. The results show that the heat capacity of nanofluids

increases significantly with an increase in the concentration of I-CNTs. The results and the trends of the enhancement of the CP_{nf} for I-CNTs are similar for non-impregnated CNTs. It can be conclude that, no effect of iron nanoparticles on the thermo-physical of the nanofluid.







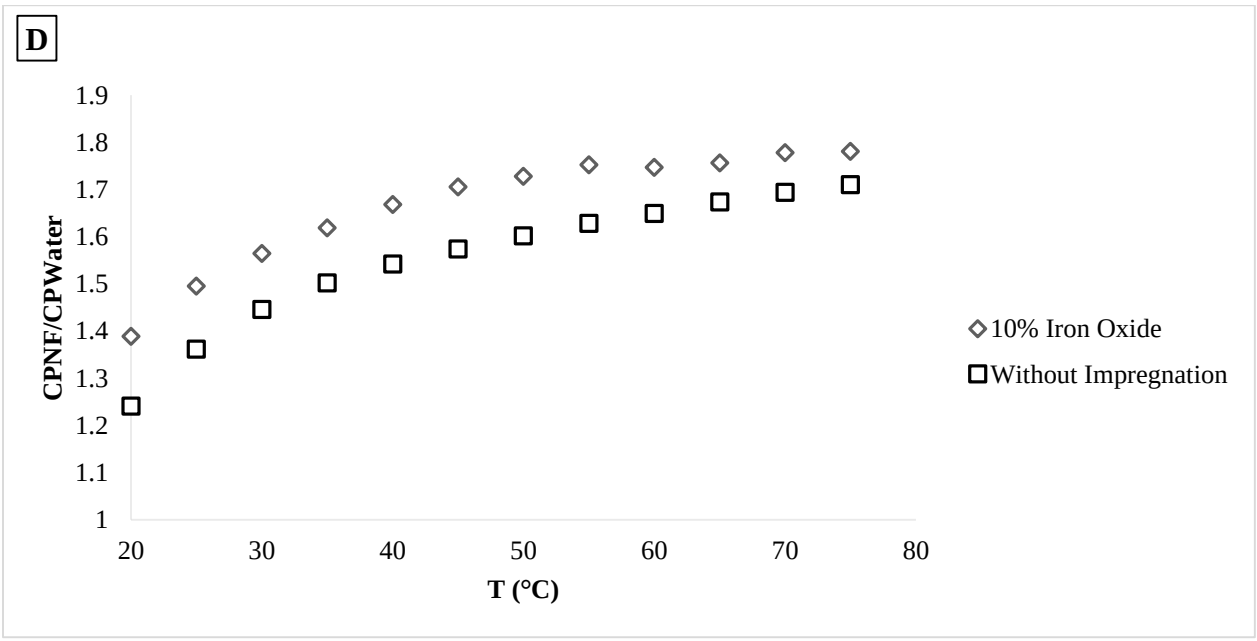


Figure 4:18: (A, B, C, D): Specific heat capacity of nanofluids as a function of temperature at loading of 10wt% doped CNT and raw CNT and different CNT loading 0.1, 0.3, 0.5 and 1 wt% using 1:0.5 GA.

CHAPTER 5

CONCLUSION

The enhancement in heat transfer characteristics such as thermal conductivity, convective heat transfer coefficient and heat transfer rate have taken much attention since discovering nanoparticles. By suspending high thermal conductivity nanoparticles in the base fluid, heat transfer properties of the new nanofluid will higher than its base fluid. Carbon and its derivatives (CNT, graphite and graphene) have taken much attention compared with other nanoparticles due to the ultra-high thermal conductivity of these particles. Nanofluids suffers from agglomeration and sedimentation of nanoparticles that mean a poor stability of nanofluids. Using surfactants is the best solution to have a long time stability of nanofluids.

Many surfactants are available in markets and used such as CTAB, PVP, SDS and GA, but the effect of each surfactant on the heat transfer characteristics and stability of the nanofluids still not studied. Surfactants also directly affected the viscosity of the nanofluids which in turn will affect pressure drop across the heat exchanger equipment. Another factor may enhance dispersion of nanoparticles and hear transfer characteristics is the impregnation of CNT with metal oxides.

In this study, we concentrated on (a) studying the effect of the surfactants GA, PVP and SDS on the stability of CNT-water nanofluids with different CNT:Surfactant ratio, (b) investigating the effect of mentioned surfactants on the thermo-physical properties of nanofluids such as density, viscosity, thermal conductivity and specific heat capacity, (c)

application of the best nanofluid properties in shell and tube heat exchanger to study the enhancement in heat transfer rate, overall heat transfer coefficient and pressure drop within the heat exchanger, and (d) studying the effect of doping iron oxide on the surface of CNT on thermo-physical properties of the nanofluids.

Previous studies in this area were concerned with the enhancement in thermal conductivity and convective heat transfer coefficient. The enhancement in thermal conductivity was not stable for all studies, enhancement ranging from (12.4 – 38 %) at concentrations less than 1vol% for [26, 28, 29, 41, 45] and other researchers, sometimes enhancement out of this range such as Rashmi [33] who got 250% enhancement in thermal conductivity at 0.1wt%. However, still no studies performed on specific heat capacity and surfactant effect on heat transfer characteristics. This thesis will concern about the missing parameters from the literature.

The effect of the three surfactants (PVP, GA and SDS) on stability of nanofluids have been performed at different CNT:Surfactant ratio and different CNT loading in the base fluid, the results shown that 1:0.5 ratio is enough to get a long time stability for more than 6 months for GA and PVP, for SDS 1:0.5 was enough to get stable solution for 3 months only. Using 1:1 ratio also found to be stable solutions for more than 6 months for the three surfactants. Samples were also tested under heating and mixing, while heating SDS produce explosive bubbles that excluded it firstly form the use in the heat exchanger. Also SDS have high adhesion forces with glass beakers which was not found with PVP and GA.

CNT that was used for sample preparation was characterized using SEM, TEM and TGA, as shown from SEM and TEM results the diameter of the CNT 10 – 20 nm and 10 – 30 μm

length, from TGA results it can be shown that CNT started to burn at 550 °C and completely burned at 650 °C.

Viscosity was also investigated to study the effect of surfactant and CNT loading. Viscosity was directly proportional to CNT and surfactant concentration. Viscosity measurements was the highest for SDS and the lowest was for GA results as shown in table 5.1. It can be concluded from viscosity measurements that at low concentration of CNT loading (i.e 0.1wt%) there is no effect of CNT:surfactant ratio but at concentration 0.5 wt% and higher, CNT:surfactant ratio is significantly affected the viscosity measurements as shown in table 5.2. Increasing in viscosity explained by the addition of surfactant and also nanoparticles has an effect on viscosity. The temperature effect on the viscosity of nanofluids also was studied, as shown from the results, the viscosity was decreased with increasing temperature and this could be explained that by increasing temperature, kinetic energy of the particles increases which in turn will decrease the viscosity also the atomic bonding between particles tend to be weaker which decreases the viscosity as the temperature increased.

Table 5:1: The percentage of increasing in viscosity for different surfactants at different CNT loading

Example: At 20 °C			
	GA	PVP	SDS
0.1 wt%	11 %	12 %	74 %
0.3 wt%	18%	26 %	198 %
0.5 wt%	30 %	38 %	290 %
1 wt%	1600 %	1700 %	ND

Table 5:2: The percentage of increasing in viscosity for different surfactant ratio at different CNT loading

Example: At 20 °C			
CNT: Surfactant	AG	PVP	CNT Loading
(1:0.5)	11 %	12 %	0.1 wt%
(1:1)	11 %	12 %	
(1:0.5)	30 %	38 %	0.5 wt%
(1:1)	65 %	80 %	

We have also investigated the effect of CNT: Surfactant ratio and CNT loading on thermal conductivity of nanofluids. There was no effect of the type of surfactant on thermal conductivity also there was no effect of CNT:Surfatant ratio on the measurement of thermal conductivity. The only factor that has significant effect on thermal conductivity is the CNT concentration (CNT loading), as shown from the results, thermal conductivity was increased with increasing CNT concentration and increasing temperature. One of the important conclusions is, the enhancement in thermal conductivity at higher temperature was more significant compared with lower temperatures. The enhancement in thermal conductivity up to 36% at 1 wt% of CNT and 45 °C. This enhancement could be explained by the motion of the nanoparticles (Brownian motion) also by the effect of dispersing ultra-high thermal conductivity nanoparticles.

The effect of surfactant ratio and CNT concentration on density also investigated. There was negligible increase in density for nanofluids compared with the base fluids. The increasing in density with CNT concentration also could be neglected. The maximum

increase in density was 0.5 % for 1wt% of CNT. This could be explained because of the minute quantities of nanoparticles used.

Specific heat capacity was also tested to study the effect of surfactant and CNT concentration. It can be shown from the results that heat capacity was significantly increased with temperature and at high temperature the enhancement will be more significant compared with low temperatures. Comparing the three surfactant with each other, SDS has the highest heat capacity compared with PVP and GA. Heat capacity for PVP and GA are close to each other but still PVP has a slight increase than GA. Comparing different ratios of CNT: Surfactant, it also can be shown that 1:1 measurements are better than 1:0.5 measurements, that could be explained because of the effect of surfactant which increases the dispersion of CNT in the base fluid. Also it can be concluded that at low temperature, the enhancement in heat capacity was approximately same for all concentrations and as temperature increases the variation will be much clear. The maximum enhancement was 88 % for 1wt% and 75 °C using (1:0.5) of PVP. The enhancement in specific heat capacity could explained by the effect of adding nanoparticles that have ability to store more energy than the base fluid. Another reason is the nano-layer that means, CNT nanoparticles will act as energy-bridge to transfer heat within the base fluid.

Also we have investigated the heat transfer enhancement in shell and tube heat exchanger by applying the best surfactant characteristics. GA was chosen for the application because it has the lowest viscosity compared to PVP and SDS. SDS was excluded from the application because of the significant viscosity and the formation of foam and the formation of explosive bobbles at high temperature. PVP has a closed properties to GA but GA has

lowest viscosity and has the lowest price. In addition of studying heat transfer enhancement also pressure drop was studied to investigate the effect of nanofluid on the pressure drop. In the heat exchanger nanofluid was the hot fluid and temperature was set 40 °C and the cold fluid is distilled water at 25 °C and 1:0.5 was used as CNT:Surfactant ratio. Heat transfer rate was increased with increasing Reynolds number and CNT loading. Enhancement in heat transfer rate in range (100 – 500 kg/hr) was (50.9 – 53.9 %) for 0.1 wt%, (55 – 61 %) for 0.3 wt% and (57 – 65.5 %) for 0.5 wt%. Even at low Reynolds the enhancement in heat transfer rate more than 50 %. Also the overall heat transfer coefficient enhanced with Reynolds, similar enhancement of heat transfer rate was noticed fir overall heat transfer coefficient. This enhancement could be explained due to the Brownian motion, the effect of suspending high thermal properties nanoparticles and the effect of coating the internal surface of the tubes with the nanofluid which enhanced the heat transfer rate across the tube side.

Pressure drop also investigated for the nanofluids, pressure drop increased with increasing Reynolds and CNT loading. The percentage of increasing pressure drop compared with the base fluid was higher at low flow rate and Reynolds. At higher Reynolds, pressure drop tend to be more close to the pressure drop of pure water. This could be explained that, as the flow becomes more fully developed as the fluid could be treated as one phase flow. The maximum increasing in pressure drop was 15 % for 0.5 wt% and 100 kg/hr but at same concentration and 500 kg/hr that percentage decreased to 9 %.

The last part that was investigated in this thesis is the effect of doping iron oxide on the thermo-physical properties of nanofluids (density, viscosity, thermal conductivity and specific heat capacity). Iron oxide was doped on CNT surface with two percentages 1%

and 10%. SDS, PVP and GA with 1:0.5 ratio were used for sample preparation. The impregnated CNT was characterized using SEM, EDX and TGA. It can be shown from the results and SEM images that there are shiny particles the CNT which want found in the raw CNT. Also TGA was used to investigate the percentage of iron oxide which found to be closely to the prepared samples.

Same measurements for density, viscosity and thermal conductivity were obtained. Adding iron oxide to the surface of CNT lowers specific heat capacity for PVP and SDS samples. For GA samples, specific heat capacity measurements for 10% of iron oxide was higher than the raw CNT. At 40 °C the percentage increase $[CP_{NF}/CP_{Water}]_{\text{Raw CNT}} - [CP_{NF}/CP_{Water}]_{\text{Impregnated CNT}}$ was 3.3%, 2.5%, 2.9% and 12.6% for 0.1, 0.3, 0.5 and 1 wt% of CNT loading. This enhancement was significant at high concentration of CNT (1 wt%). This enhancement due to good adhesion forces between the iron and GA which acts as a double barrier of heat transport.

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